METHODS FOR THE

RESTORATION OF OBLITERATED SERIAL NUMBERS

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This handbook contains detailed information on the techniques of the criminal investigator in the performance of serial number restorations. The information should be treated as classified. Distribution of this handbook is restricted to authorized personnel only.

PREFACE

A frequent task for the crime laboratory investigator is the restoration of serial numbers or other markings which have been obliterated from metal objects. Handguns are the most common of these objects, but the investigator may also encounter rifles, shotguns, motor vehicles, bicycles, cameras, appliances, and jewelry as restoration specimens.

For the last forty years various restoration methods have been reported in the criminal science literature. Most often described are the chemical and electrolytic methods wherein the specimen surface is polished and etched in order to recover the number. A nondestructive method involving magnetic particles and a method in which the specimen is heated to restore the number are also well documented.

In 1973 Stanley G. Young of the NASA Lewis Research Center announced a new restoration method that uses the etching action of water in a state known as ultrasonic cavitation to effect restoration. Responsibility for development and evaluation of the ultrasonic method was contracted by NASA to Chicago State University in 1974. In the early stages of the project it became evident that no study had ever been published in which restoration methods were evaluated under scientifically controlled conditions. Therefore, the role of the project was expanded to include the design of a test suitable for measuring restoration effectiveness and the evaluation of all known restoration methods according to this test. Prior to the final evaluation each method was tested with variations in procedure in order to determine its conditions for optimum effectiveness.

This handbook reports the results of the research work at Chicago State University and is intended to serve as a convenient reference for the crime laboratory investigator. It begins with background information relevant to serial number restoration work (Chapter 1) and continues with a description of the general procedures adopted in the project and the theory upon which successful restorations are based (Chapter 2). A major portion of the

handbook is devoted to reporting results on the optimization of each method (Chapters 3 through 6). Specimens used for this purpose were fabricated in the laboratory from eight metals chosen to represent those most commonly encountered in the crime laboratory. Final evaluation of each method (Chapter 7) was done on these laboratory specimens and also authentic specimens, particularly handguns. The handbook concludes with specific recommendations to the investigator concerning preferred restoration methods (Chapter 8).

There is no reason to suppose the limit in restoration effectiveness has been reached with the procedures recommended herein. Hopefully, the present work will encourage further research on the subject. The author invites inquiries, suggestions and reports of new findings from other investigators.

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CHAPTER 1
BACKGROUND

INTRODUCTION

The restoration of obliterated serial numbers and other stamped markings on metals has become a major function of the modern crime investigation laboratory. The Chicago Police Department Criminalistic Division, alone, performs over 500 restorations per year on confiscated firearm serial numbers (1). Restorations on other specimen types, such as automobiles, motorcycles, and bicycles are performed by different investigating units in this department.

Restoration of a number can be accomplished whenever the obliterating process has not totally removed all evidence of the number. Because the stamping of a number deforms the metal's crystalline structure well below the indentation, a number can appear to have been obliterated while much evidence still remains. Figure 1-1 shows the deformation existing below two handgun serial numbers. Any experimental technique that can distinguish deformed from nondeformed metal is potentially a restoration method. Such techniques are well known in the fields of metallography and metallurgy. Their applications to recovering obliterated numbers have been reported by criminal investigators in the United States and Europe for at least forty years.

In 1940 G. W. Pirk, a consulting metallurgist for the Bureau of Police, Utica, New York, suggested that a chemical etching solution known to metallographers as Fry's reagent could be used to recover serial numbers (2). Since that time many additional chemical etchants have been recommended by other investigators. Particularly noteworthy among reports of the chemical method are the works of Mathews and Nicholls (3, 4).

The Federal Bureau of Investigation in 1950 reported a nondestructive restoration method based upon the behavior of magnetic particles on the

specimen surface while it is magnetic (5). At about this same time a variation of the chemical method was devised in which an electric current is used to facilitate the etching process. This electrolytic method was reported by Turner, Arai, and Mathews (3, 6, 7). The FBI has also developed a technique based on heating the specimen with a torch (8). Most recently, a novel etching method has been demonstrated by Young of the National Aeronautics and Space Administration. It uses water agitated by a vibrator into a state of ultrasonic cavitation to accomplish the etching (9). All of these methods, and several more which have been proposed but not well documented, are described in detail in later chapters of this handbook.

The method most commonly used in criminal laboratories today is probably the chemical method. It is considered to be relatively effective and requires only the simplest of equipment. Prior to the work described in this handbook there never appears to have been a study which scientifically evaluated the various methods for effectiveness.

SERIAL NUMBERS AND LAW ENFORCEMENT

A comprehensive treatment of the role of serial numbers in law enforcement is beyond the scope of this work. A brief account is given, however, as relates to the subject of serial number restoration.

A significant step in the regulation of firearms in the United States was the federal Gun Control Act of 1968. According to this legislation all weapons manufactured or imported must bear a readily visible serial number located on the frame or receiver. To maintain the integrity of the number the law states:

> No person shall knowingly transport, ship, or receive in
> interstate commerce any firearm which has had the
> importer's or manufacturer's serial number removed,
> obliterated, or altered.

Similar laws pertaining to items other than firearms are commonly legislated by local governments. For example, an ordinance of the City of Chicago makes it a felony for:

> A person to remove, alter, deface, destroy of falsify a
> manufacturer's identification number of a vehicle or an
> engine number of a motor vehicle or any component part
> thereof having an identification number.

With respect to bicycles a City of Chicago ordinance states:

> It shall be unlawful to destroy, remove, alter, cover or
> deface the manufacturer's serial number on any bicycle.
> It shall be unlawful for any person to own or have custody
> of a bicycle, the original manufacturer's serial number
> of which has been destroyed, removed, altered, covered
> or defaced. Any person who violates any of the provisions
> of this section shall be fined not more than two hundred
> dollars for each offense.

The probability that the ownership of a firearm, vehicle, or other object can be traced through its serial number depends upon the regulations governing registration and serial number record keeping. There is no federal handgun registration law in the United States. According to the Gun Control Act of 1968, licensed gun dealers must require each buyer to produce identification and complete a form designed to exclude dangerous persons from purchasing firearms. Once the purchase has been made, the weapon may legally change ownership with no record required. In the United States registration of motor vehicles is the responsibility of the individual states. Some municipalities register firearms and bicycles. For the purpose of tracing numbers law enforcement personnel may direct inquiries to the National Crime Information Center. This agency collects data on serial numbers on stolen firearms and vehicles. Other suggested sources of information are manufacturers, retailers, pawnbrokers, repair shops, and insurance companies (10).

It is only the number appearing on the frame of a firearm which is used for its legal registration. Many manufacturers follow the practice of repeating this number at other locations on the weapon. These hidden numbers can be of use to the investigator if the frame number has been obliterated. For

obvious reasons caution must be exercised in drawing conclusions about the registration of the weapon from any number found on an interchangeable part. Some manufacturers follow the practice of placing the last two or three digits of the serial number at hidden loactions on the weapon. These partial serial numbers can be useful if complete restoration of the frame number cannot be accomplished, but they should not be confused with other numbers sometimes used by manufacturers to identify specific models, parts, or assembly inspectors. Common locations of serial numbers on firearms are illustrated in Figure 1-2.

Detailed information on serial numbering systems, including extensive tables and photographs showing the location of hidden numbers for more than five hundred handguns, can be found in the reference work by Krcma (11). A briefer account is also available by the same author (12). Firearm manufacturers can also be contacted by qualified persons for such information.

Automobiles and bicycles have Vehicle Identification Numbers (VIN) on frames, motors, and often other locations. A typical VIN consists of 13 numbers or letters coded to give information on the manufacturer, body and engine type, assembly plant, model year, and sequential production number. The National Automobile Theft Bureau publishes the Manual for the Identification of Automobiles, which contains information on the location and interpretation of these numbers. Recently-built automobiles have the VIN stamped on a metal plate visible through the windshield. It is reported that parts such as transmissions and crankcases sometimes bear their own unique serial numbers and that manufacturers' records cross-reference these to the vehicle numbers (13).

Examination of the serial number restoration case load of a representative police department gives insight into practical aspects of the restoration problem (1). In 1974 the Criminalistics Division of the Chicago Police Department was requested by its investigators to perform restorations on 530 firearms. In this department a restoration is considered successful if the frame number is completely recovered or if enough is made legible to establish it to be the same as a hidden number found elsewhere on the firearm.

Table 1-1 itemizes the restoration results for 124 firearms examined by the Criminalistic Division in the first quarter of 1974. The specimens are categorized according to whether the frame is of steel or, as in the case with less expensive firearms, of aluminum or zinc alloys. Approximately one third of the firearms fall into the latter category. A total of 75 firearms of this sampling were successfully restored. The success rate was 63% for steel frames and 54% for aluminum or zinc alloy frames. All serial numbers restored by the Criminalistic Division are checked for listing by the National Crime Information Center. Of the 75 restorations only 10 were so listed. Similarly, a check is made for registration of the firearm with the City of Chicago. Only four of the firearms were found to be registered; none of them were in the aluminum or zinc alloy frame category. Clearly, the contribution to law enforcement made by serial number restoration work would be greatly enhanced by more comprehensive registration laws.

FIREARM MANUFACTURING AND SERIAL NUMBERING METHODS

Within the firearms industry there is little uniformity with respect to the metal alloys and fabricating procedures used. Each manufacturer has preferred materials and techniques, and these may differ for each firearm model produced. Even in the case of a single model made by one company over a period of years, variations in manufacturing may occur with time.

Specific information on manufacturing methods is often regarded as classified by members of the industry. For this reason no reference works are available on the subject, although such would be useful to the investigator of firearm serial numbers seeking complete understanding of the specimen. The present section briefly describes manufacturing procedures and alloys used for selected firearms and is based upon information supplied by the respective manufacturers. For helpful background information on alloy designations, metallurgical processes, and finishes applied to firearms, the reader is referred to a book written for the amateur gunsmith by MacFarland (14) and to standard metallurgy books.

Table 1-2 lists selected firearms typical of those commonly found in the United States. The firearms are categorized according to the metal alloy

used for making the frame (or receiver, in the case of rifles and shotguns), since this part carries the principal serial number.

Included in the categories of metals listed in Table 1-2 are the low- and medium-carbon steels. Carbon steels are distinguished by the fact that the chief alloying element is carbon. Other elements are not added in appreciable amounts. These steels have AISI (American Iron and Steel Institute) designations 10XX or 11XX; the latter category indicates a resulfurized metal. The last two digits in these designations indicate the carbon content in hundredths of one percent. Low-carbon steels have 0.25% or less carbon and medium-carbon steels have 0.25 to 0.50% carbon.

Carbon steels have a long history in the manufacture of firearms, but modern times have also seen the introduction of alloy steels having special properties. Alloy steels may be considered as carbon steels to which have been added elements to enhance desired characteristics. Typically, the total percentage of alloying elements does not exceed 5% for this classification. Chromium and molybdenum commonly are used to improve hardenability and resistance to corrosion. AISI designations 40XX and 41XX indicate carbon/molybdenum and chromium/molybdenum alloys, respectively. High-cost firearms having even greater corrosion resistance are made of stainless steel. The chromium content of stainless steels may be as high as 27%. Nickel is frequently also present in stainless steel.

Low-cost firearms often have frames of aluminum alloy or zinc alloy. AA (Aluminum Association) series 2XXX and 7XXX indicate aluminum/copper and aluminum/zinc alloys, respectively. These are forging alloys possessing the high strength desired for firearm manufacturing and the light weight characteristic of aluminum. The most common zinc alloy contains about 4% aluminum and lesser amounts of copper and magnesium. It is not as lightweight or soft as the aluminium alloys.

Manufacturing procedures typically used in the manufacture of high-quality firearms are illustrated by the various handgun models made by Colt Industries of Hartford, Connecticut (15). Many Colt revolvers have frames of alloy steel, including the Trooper, Detective, Law Man, and Python models.

The Trooper is made from hot rolled AISI 4040 modified alloy, which is forged and then machined to specifications. The frame is serial numbered while the metal is in the normalized state. It receives no further heat treatment or hardening after numbering. As a final step a protective finish of gun blue (conventional black oxide) or nickel electroplate is applied. As is often the case, this gun is made from more than a single metal; its slide plate is of AISI C1018 low-carbon steel. The slide plate and frame carry the same serial number.

Colt Single Action Army .45-caliber revolvers and also the 1700 and 2400 series models are made of medium-carbon steels such as AISI 1026 and 1137. The frame is forged and machined, and is in the normalized state when the serial number is applied. For these steels the hardness and durability required for a handgun is achieved by color case hardening as a final step.

The Lightweight Commander .45 ACP pistol made by Colt has a frame forged from wrought aluminum alloy. After the piece has been shaped, it is serial numbered and receives no further treatment except for a blue (sulfuric acid soft anodized) or nickel electroplate finish.

RG Industries of Miami, Florida produces revolver frames from both zinc alloy and steel (16). In either case the piece is cast rather than forged. The Model-40 is a .38 Special (Spc.) revolver having a die cast zinc alloy frame. After casting, the part is machined and serial numbered. It is blued as a finishing step. The Model-88 is a .357 Magnum (Mag.) revolver with a frame investment cast from carbon steel. It is in the annealed condition when serial numbered and also receives a gun blue finish.

TECHNIQUES COMMONLY USED TO OBLITERATE NUMBERS

A variety of techniques are used by criminals to unlawfully obliterate serial numbers and sometimes to conceal the fact that this has been done (1, 10, 13). Certain of these techniques make the restoration task more difficult.

Scratching with Sharp Tool or Filing.—This crude technique is effective on softer metals, particularly aluminum alloy. The surface may be in a rough condition and may require considerable smoothing before

restoration treatment can begin.

Grinding with Power Tool. — Occasionally this process is performed with great care and precision in an effort to make the obliteration less obvious. The amount of metal removed can vary, and so does the difficulty encountered in accomplishing the restoration.

Peening with Hammer. — Cold-working the metal (see Chapter 2) by hammering over the area of an obliterated number has the effect of masking the deformed metal evidence upon which a successful restoration is based. This technique has been encountered by investigators more frequently in recent years, possibly because its effectiveness is becoming known (1).

Overstamping and Overpunching. — In the first of these procedures a false serial number is applied over the area of a previously obliterated number. Both this process and overpunching with a sharp tool have the same effect of cold-working the metal and masking evidence as does peening.

Welding and Other Heating Processes. — Adding fresh metal to the surface by welding results in obvious complications. Heating alone also causes problems in that it has the effect of normalizing or annealing metal and thus destroying the disturbed metal evidence.

Rusting. — This naturally occurring process can result in the unintentional obliteration of a serial number. All ferrous metals with the exception of stainless steel are subject to rusting. Finishing techniques such as gun blueing and nickel or chrome plating retard rusting but do not eliminate it. Rust must be removed before a restoration can be accomplished.

Reapplication of the Original Finish. — In an effort to enhance the appearance and resale value of a firearm or to conceal the fact that serial number has been obliterated, the offender may reapply the original finish of the weapon. A gun blue finish applied over an obliteration typically creates no problem for a restoration effort. Nickel or chrome plating, however, must be removed prior to restoration work.

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Table 1-1

Serial Number Restorations on Firearms by the Chicago Police Department Criminalistics Division in January-March 1974

	Total firearms	Successful restorations	NCIC[a] listed	City registered
Steel frame revolvers				
Smith & Wesson .38-caliber	17	16	3	1
Colt .38-caliber	8	6	0	0
H & R .32-caliber	7	3	0	0
Colt .32-caliber	4	2	0	0
Iver Johnson .38-, .32-, and .22-caliber	4	2	1	1
All others	17	8	0	0
Steel frame pistols				
Colt .45- and .32-caliber	4	4	1	1
Ruger .22-caliber	3	3	1	0
Remington .45-caliber	2	0	0	0
All others	1.	8	1	1
Steel receiver rifles	4	1	0	0
Steel receiver shotguns	3	2	0	0
Aluminum or zinc alloy frame revolvers				
Clerke .32-caliber	8	3	1	0
RG .22-caliber	7	6	1	0
RG .38-caliber	3	2	0	0
Rohm .22-caliber	6	6	1	0
All others	6	1	0	0

Table 1-1 (Cont.)

	Total firearms	Successful restorations	NCIC [a] listed	City registered
Aluminum or zinc alloy frame pistols				
Titan .25-caliber	4	1	0	0
All others	3	1	0	0
Totals	124	75	10	4

[a]National Crime Information Center

Table 1-2

Firearms Classified by Metal Alloy Used in Frame

Manufacturer	Models	Specifications[a]
Low-Carbon Steel Frames		
Garcia (F.I. Industries)	Brazilian-made revolvers	AISI 1020, forged
Winchester-Western	Model-9422 rifle	AISI 1117
Medium-Carbon Steel Frames		
Benan	.25-caliber auto. pistol	- - -
Browning	Japanese-made rifles	AISI 1045 and 1046
Colt	Single Action Army revolver	AISI 1026, forged
	Series 1700 and 2400 revolvers Model-S .22 L.R. auto. pistol	AISI 1137, forged
Ithaca	Model-37 shotgun	AISI 1130
	Model-51 and Mag-10 shotguns	AISI 1040
Mauser (Interarms)	Parabellum auto. pistol	Carbon steel, forged
RG Industries	Model-88 .357-Mag. revolver	AISI C1043, investment cast
Virginian (Interarms)	Single Action .357-Mag. revolver .45-caliber revolver	- - -
Winchester-Western	Super-X Model-1 shotgun	AISI 1141
	Model-101 shotgun	AISI 1040
Alloy Steel Frames		
Colt	Trooper .357-Mag. revolver	AISI 4040
	Detective .38-Spc. Model-D revolver Law Man and Police Python .357-Mag. Model -I revolvers	AISI 4140

Table 1-2 (Cont.)

Manufacturer	Models	Specifications[a]
Garcia (F.I. Industries)	Model-D .380-caliber pistol	AISI 4140
High Standard	Target pistols Recent-made revolvers	AISI 4140
Llama (Stoger Industries)	Auto. pistols Martial revolver	AISI 4140
Ruger	Model-108 .38-Spc., Model-107 .357-Mag., and Blackhawk revolvers	- - -
Winchester-Western	Model-70, 70A, and 52 rifles Model-12 shotgun	AISI 4140

Stainless Steel Frames

Ruger	Model-717 .357-Mag. revolver	- - -
Security Industries	Police Security .38-caliber, Security Undercover .357-Mag., and Police Pocket .357-Mag. revolvers	- - -
Smith & Wesson	Chiefs Special Model-60, Military and Police Model-64, Combat Magnum Model-66, and K-38 Combat Model-67 revolvers	- - -
Sterling Arms	Model-400S .38-caliber, 300S .25-caliber, and 302S .22-caliber pistols	- - -

Aluminum Alloy Frames

Colt	Lightweight Commander .45 ACP pistol Model-D Lightweight .38-Spc. revolver	AA 2014-T6, forged
High Standard	Sentinel .22-caliber revolver	- - -
Remington	Model-552 and 572 .22-caliber rifles	- - -
Luger (Stoger Industries)	.22-caliber auto pistol	AA 7075-T6, forged

Table 1-2 (Cont.)

Manufacturer	Models	Specifications[a]
Winchester-Western	Model-1200 and 1400 shotguns	AA 2014-T6 and 7075-T6
Zinc Alloy Frames		
Ithaca	Youth Model .22-caliber rifle	Zamac
RG Industries	Model-40 .38-Spc. revolver	ASTM AC41A, die cast
Titan	Tiger .38-Spc. and .25-caliber auto. pistols	- - -

[a]AISI: American Iron and Steel Institute
AA: Aluminum Association
ASTM: American Society for Testing and Materials
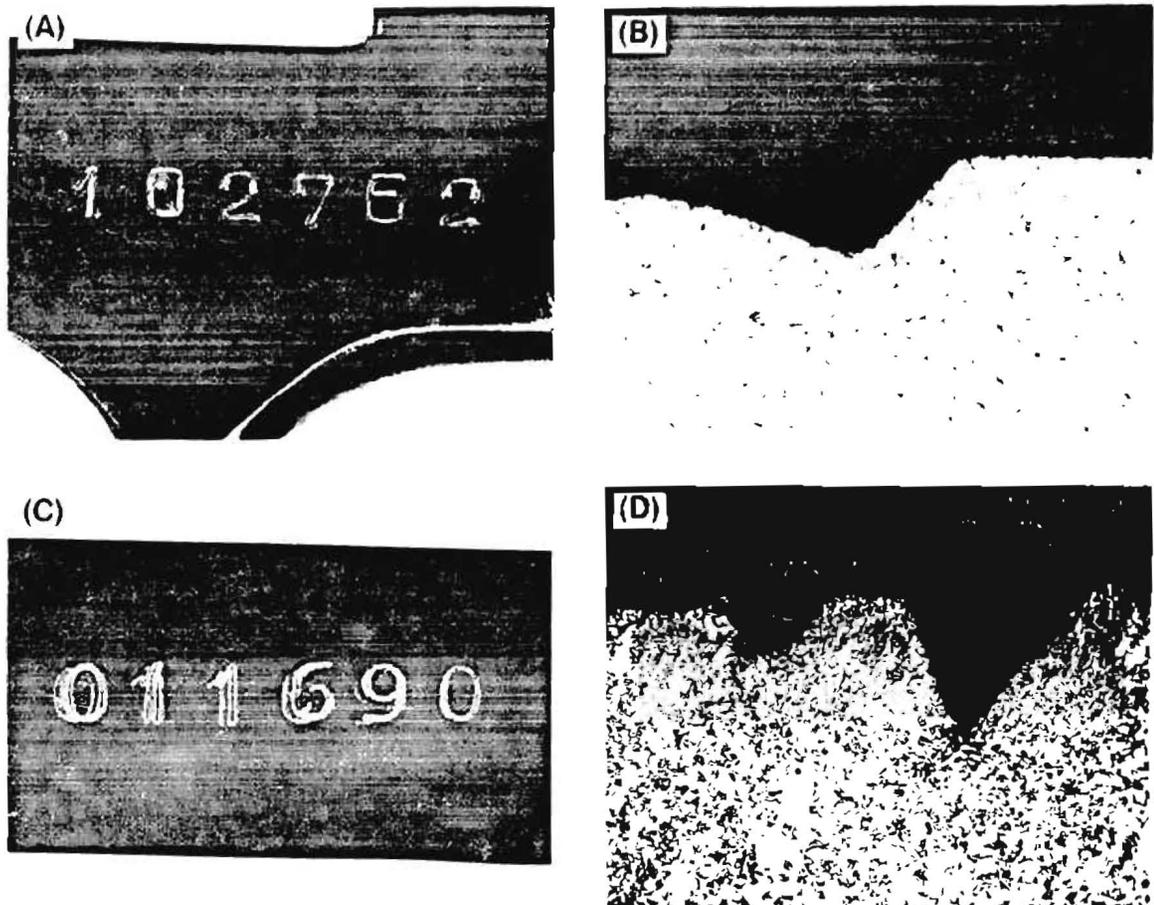
Figure 1-1. Handgun serial numbers and their metallographic cross-sectional views. (A) Rossi .38 Spc. steel frame revolver and (B) Cross section (100X) of numeral on this frame (2 nital etchant). (C) RG 40 .38 Spc. zinc alloy frame revolver and (D) Cross section (100X) of numeral on this frame (Palmerton's etchant).

(A)

(B)

(C)
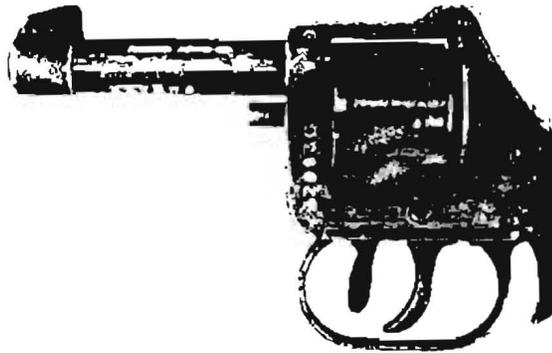
(D)

(E)

(F)

Figure 1-2. Common locations of serial numbers on firearms. (A) Revolver butt (B) Cylinder (C) Barrel and frame (D, E) Pistol frame. (F) Shotgun frame. (Shotgun photo courtesy Winchester-Western.)

CHAPTER 2
GENERAL EXPERIMENTAL PROCEDURES AND
THEORETICAL CONSIDERATIONS

PREPARATION OF LABORATORY SPECIMENS

Most restoration testing reported in this handbook was done on numbered specimens prepared in the laboratory specifically for that purpose. Specimens were made from eight different metals, five ferrous and three nonferrous. The metals, chosen to be representative of those most often encountered in crime laboratories, are listed in Table 2-1 along with their fabricating descriptions, Brinell hardnesses, densities, and uses. Their chemical compositions are given in Table 2-2.

Laboratory specimens were prepared from pieces of test metals approximately 3 cm square. The initial thickness of each piece was measured by a micrometer. Each piece was then stamped with a single number by use of a hardened steel die (see Figure 2-1). The numbers were 8 mm high and were stamped to a depth of approximately 0.3 mm. To accomplish a consistent stamping depth for a given metal, the die was struck with a 1.4-kg hammer dropped from a fixed height, which varied with the hardness of the metal. The stamping depth of the number was measured with a gauge as shown in Figure 2-1.

Obliteration of numbers was accomplished by uniform grinding of the specimens on a belt sander using medium (60-grit) silicon carbide paper. After the desired amount of metal was removed, the thickness of the specimen was again measured. The thickness of metal removed could then be calculated as the difference. Typically, the thickness of metal removed was greater than the depth of the original number. Throughout this report the extent of grinding is expressed as removal depth, the ratio of the thickness removed to the stamping depth of the number. This quantity is defined by the following equation:

$$\text{Removal depth} = \frac{\text{Initial thickness} - \text{Final thickness}}{\text{Stamping depth}}$$

For example, a removal depth of 1.50 indicates that grinding was performed until the thickness of metal removed was 1.50 times the depth of the number itself. Figure 2-2 illustrates various removal depths by showing a cross-sectional view of a stamped number.

For preliminary testing of each restoration method, specimens were ground just to the point where the number was entirely obliterated from sight. The term just-obliterated is used to describe such specimens. A significant finding of the work is that for most of the metals just-obliteration is consistently reached at removal depths of less than 1.00. Specifically, the removal depth required to accomplish complete disappearance of the number for each metal is as follows:

Metal	Removal Depth
Alloy steel	0.88
Cast iron	0.85
Low-carbon steel	0.86
Tool steel	0.62
Stainless steel	0.67
Aluminum alloy	0.73
Brass	1.04
Zinc alloy	0.88

Values less than 1.00 result when fragments of metal produced in the grinding process become imbedded in the base of the groove of the number, which creates the impression of complete obliteration (see Figure 2-2). This observation, that smeared metal deposited in the number indentation may cause the number to appear obliterated before it is completely removed, has been reported by Young (1, 2). As might be expected, impressive restoration results can be easily obtained for a just-obliterated specimen having a removal depth less than 1.00.

Most restoration procedures require the speciman to be polished as a preliminary step in the recovery procedure. Polishing of all specimens was done on a cloth polishing wheel with white rouge applied to it. Overheating of the specimen during polishing was prevented by regular squirting with a 50% mixture of acetone in water. Polishing was continued until all scratches were removed and a mirror-like finish was attained. Polishing compound was removed by washing with hexane solvent. Specimens of

iron and steel required considerably more time for polishing than those of aluminum alloy, brass, and zinc alloy. To reduce polishing times, iron and steel specimens were given a fine grinding with 240- or 320-grit aluminum oxide cloth just before polishing.

CRITERION USED TO EVALUATE RESTORATION EFFECTIVENESS

The value of any restoration method lies in its ability to recover numbers which have been subjected to more severe grinding than that which produces the just-obliterated condition. Thus, the criterion established in this work for the evaluation of a restoration method is its effectiveness at recovering a number throughout the range of removal depths. As the removal depth increases, all methods will eventually fail to restore the number. That method which accomplishes restoration at the greatest depth is judged to be best. Figure 2-3 shows a series of photographs of a restored number on stainless steel at various removal depths.

Chapter 7 contains data concerning the percentage of an obliterated number that is restored with increasing removal depth. To obtain such data, a numbered specimen is first ground to just-obliteration and a restoration attempted by some chosen method. The percentage of the obliterated number restored to visibility is estimated visually and recorded. Assuming that at least a portion of the number is recovered, the specimen is ground further and the restoration attempted a second time. This sequence is continued until the chosen restoration method fails to recover even a portion of the number. For methods in which the restoration treatment causes destruction or alteration of the metal below the surface, the sequential procedure is not used; instead, fresh specimens are prepared at each removal depth.

METAL DEFORMATION AND ITS EFFECTS (3)

It is because of alterations which occur in the crystalline structure of a metal upon impressing a serial number that obliterated numbers can be restored. A brief description of the theory of metal deformation follows.

Microscopic examination reveals that metals are polycrystalline in structure. They consist of irregularly shaped crystals, or grains, which form when molten metal cools to the point of solidification. Between the grains are interlocking regions known as grain boundaries. Figure 2-4(A) shows the grain structure in brass which is typical of an annealed metal.

Metal atoms in the crystal grains are arranged in an orderly three-dimensional array, or space lattice. The atomic arrangement within grain boundaries is less regular than that in individual crystals, and this is believed to be the reason for the greater strength observed at grain boundaries. By regulating the cooling rate during solidification, the size of the grains and the density of the grain boundaries can be controlled. The mechanical properties of the metal are thereby affected, small grains result in greater strength and toughness, whereas large grains promote better plasticity.

When a stress (tension, compression, or twist) is applied to a metal, its grains are deformed. If the stress exceeds the elastic limit of the metal, the structure does not return to its original condition upon removal of the stress. The result is permanent deformation, also called plastic deformation. Because metals display this property, they can be shaped by such processes as rolling, drawing, bending, extruding, and forging.

Plastic deformation results in two kinds of movement of atoms within the crystal, known as slip and twinning. The mode of movement for each is illustrated in Figure 2-5. Slip involves the shearing of one block of atoms over the remainder of the lattice by some multiple of the inter-atomic distance. Such deformation occurs along specific crystallographic directions called slip planes. The process creates new edges at the crystal surface, resulting in slip lines observable under metallographic examination. Twinning results when parallel planes of atoms slip consecutively over each other by some fraction of the interatomic distance. A new lattice orientation results along twinning planes. Any crystal plane crossing the twinning planes is bent by this deformation. Thus, the region between the planes can be observed metallographically and is known as a twin band.

For a polycrystalline metal, an applied stress is transmitted through the material from one grain to another, causing plastic deformation throughout. The result is the appearance of slip lines and twin bands and a decrease in grain size. This process is illustrated in Figure 2-4, which shows a metallograph of brass subjected to cold rolling.

When a serial number is stamped or pressed into a metal, the stress created is greatest at the point of application of the die. Figure 2-6 schematically illustrates the compressive forces resulting and the plastic deformation region below the number. Beyond this localized region the compressive forces are too dissipated to cause plastic deformation, but a deeper region of elastic deformation does exist. Cross-sectional views of stamped numbers on various metals are shown in Figure 2-7.

It is well known that a bent piece of wire is difficult to straighten again. In particular, the bent portion has a special strength that resists a return to the original shape. This observation illustrates one of many ways in which plastic deformation affects a metal. Industry takes advantage of such effects to modify the properties of metals in desired ways. All fabricating processes, such as cold rolling, hammering, drawing, stamping, pressing, and bending, produce plastic deformation. Metal workers usually use the terms cold-working, work-hardening, or strain-hardening to describe the results of these processes. Even the process of machining or polishing a metal has the effect of cold-working it.

In general, all properties of a metal are affected by cold-working. Properties whose magnitudes are known to increase are:

hardness	electrical resistance
brittleness	rate of dissolution by chemicals
tensile strength	magnetic retentivity
yield strength	elastic limit

As might be expected from the above effects, cold-working <u>decreases</u>:

ductility	resistance to chemical attack
impact strength	magnetic permeability
density	plasticity

All cold-working effects are removed if the metal is annealed. In this process the metal is heated to a point where deformed structure disappears. All serial number restoration procedures are based upon the principle that the deformed or cold-worked metal immediately below the stamping has different properties than its surroundings.

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Table 2-1

Metals Used in this Study

	Standard designation[a]	Description	Brinell hardness number	Density g/cm^3	Common uses
Alloy steel	AISI 4140	Cold-drawn annealed bar; chromium/ molybdenum alloy steel; machinability 60%	207	7.85	Firearms, gears bearings, automobile parts
Cast iron	ASTM 4040	Fine-grained gray iron bar; 40,000-psi tensile strength	159	7.15	Engine blocks, appliances, pipe, electric motor frames
Low-carbon steel	AISI 1116	Free-machining bar, resulfurized plain carbon steel; machinability 91%	121	7.96	Firearms, machine parts, structural steel
Tool steel	AISI O1	Annealed bar, cold-worked tool steel for oil hardening	183	7.80	Dies, punches, gauges, bushings
Stainless steel	Type 301	Austenitic (nonmagnetic) steel fabricated as hinges	156	8.11	Firearms, household utensils

Table 2-1 (Cont.)

	Standard designation[a]	Description	Brinell hardness number	Density, g/cm^3	Common uses
Aluminum alloy	AA 6063	Extruded bar, magnesium/silicon alloy	35	2.75	Extruded hardware
Brass	ASTM B-16	Free-cutting wrought brass bar, half hard temper	134	2.75	Cartridge cases, wire, screws, machine parts
Zinc alloy	ASTM AG-40A	Cast rectangular bar, aluminum alloy	82	6.70	Firearms, automotive parts, office equipment, hardware

[a] AISI: American Iron and Steel Institute
AA: Aluminum Association
ASTM: American Society for Testing and Materials

Table 2-2

Chemical Composition of Metals of this Study

Percent of Element[a]						
C	Mn	P	S	Si	Cr	Other
0.38/ 0.43	0.75/ 1.00	<0.035	<0.04	0.20/ 0.35	0.80/ 1.10	0.15/ 0.25 Mo
3.49	0.78	0.08		2.61		0.09 Cu
0.08	1.05	0.02	0.08			
0.90	1.20				0.50	0.50W 0.20V
0.03	1.94	0.01	0.01	0.27	18.25	8.90 Ni 0.13 Mo
	0.15			0.40/ 0.80		0.80/ 1.20 Mg 0.70 Fe 0.25 Zn 0.15/ 0.40 Cu 0.15 Ca 0.15 Ti
						60.17 Cu 35.80 Zn 3.59 Pb 0.25 Sn
						3.90/ 4.30 Al <0.10 Cu <0.075 Fe

Row labels (left column, top to bottom): Alloy steel; Cast iron; Low-carbon steel; Tool steel; Stainless steel; Aluminum alloy; Brass; Zinc alloy

Table 2-2 (Cont.)

Percent of Element[a]						
C	Mn	P	S	Si	Cr	Other

0.025/
0.050 Mg

[a] Analyses by X-ray spectrograph for cast iron, low-carbon steel, stainless steel and bras; all others are standardized compositions based upon manufacturer's designation for metal.
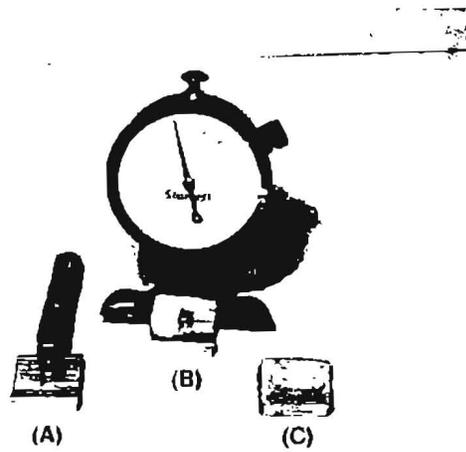
Figure 2-1. Laboratory specimen preparation. (A) Stamping die.
(B) Depth gauge. (C) Obliterated specimen.

Imbedded particles

Just-
obliterated

1.00

Removal
depth

2.00
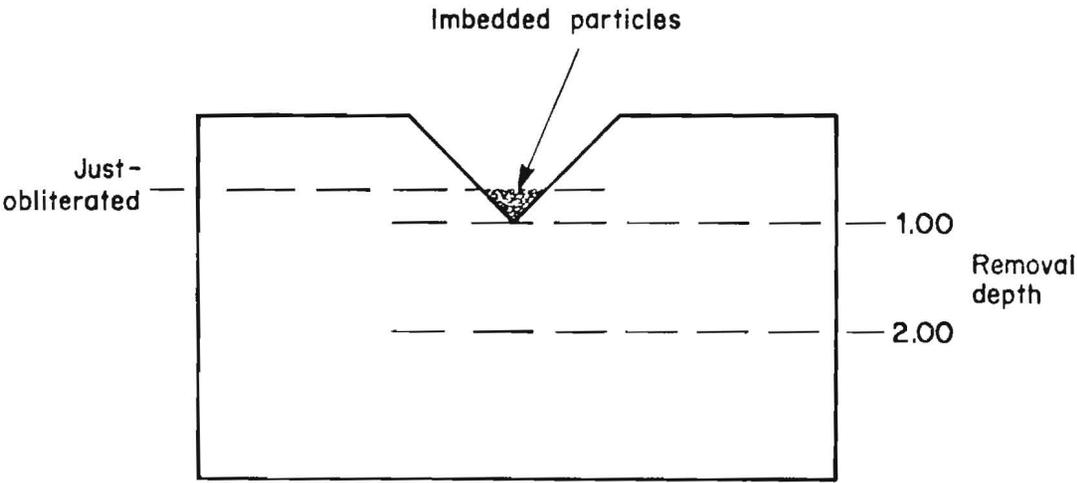
Figure 2-2. Cross-sectional view of stamped number. Removal depths of
1.00 and 2.00 are indicated; just-obliteration may occur at a removal
depth less than 1.00 due to imbedded particles.

Obliteration depth 1.01

Obliteration depth 1.45
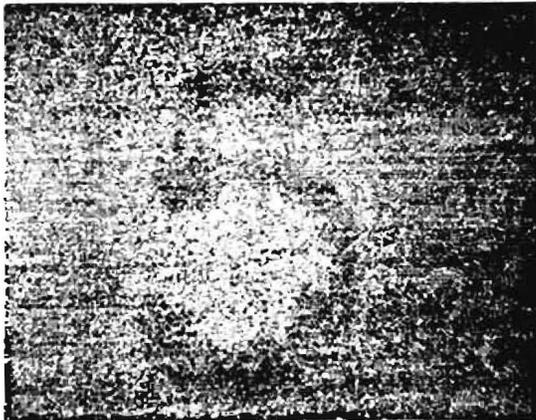
Obliteration depth 1.82
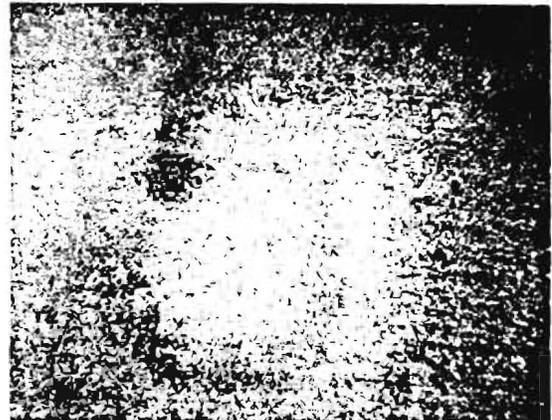
Obliteration depth 2.48

Figure 2-3. Restoration of number (8X) from various removal depths. The specimen is stainless steel and restoration is by the chemical method using Fry's reagent.
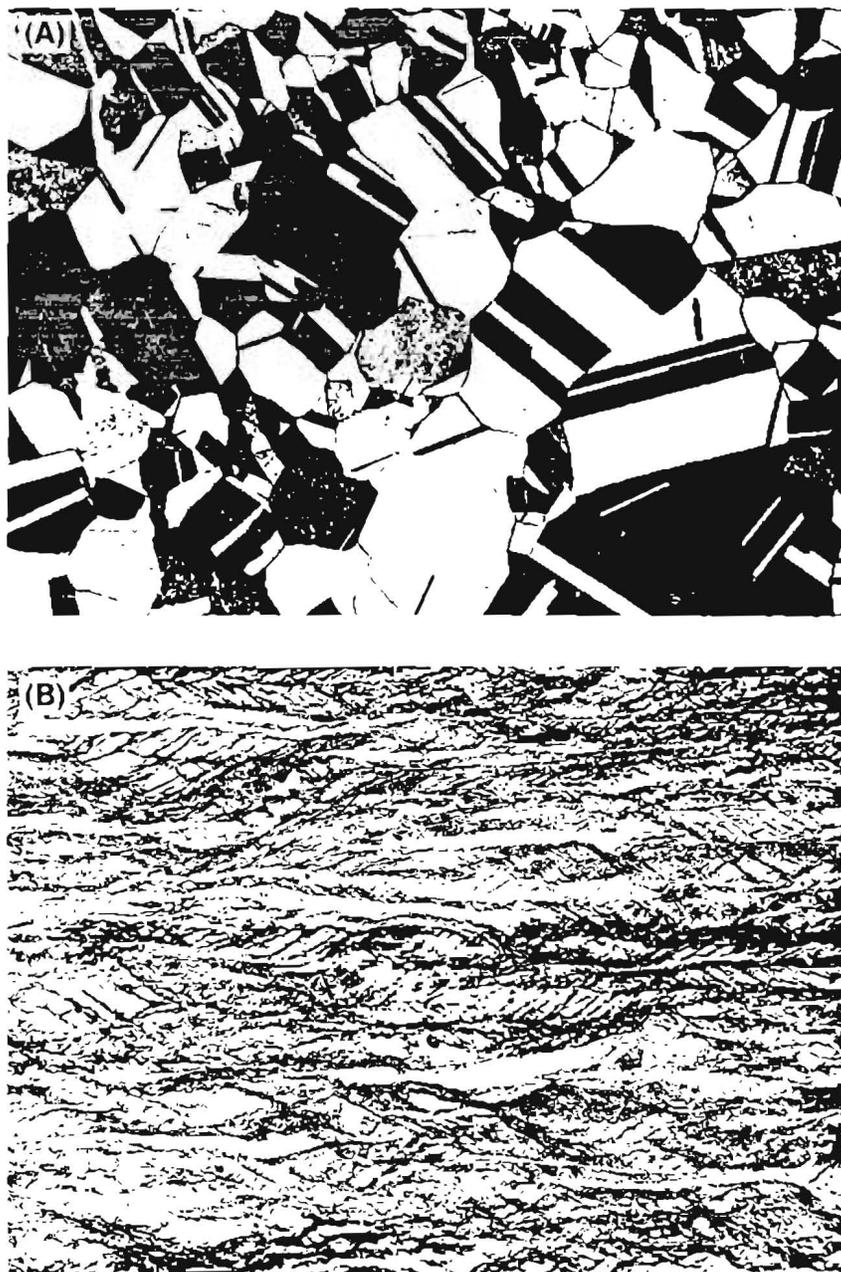
Figure 2-4. Grain structure of brass. (A) In the annealed condition. (B) After extensive deformation by rolling. Metallographs (300X) obtained with NH_4OH, H_2O etchant. (Courtesy Buehler Ltd.)
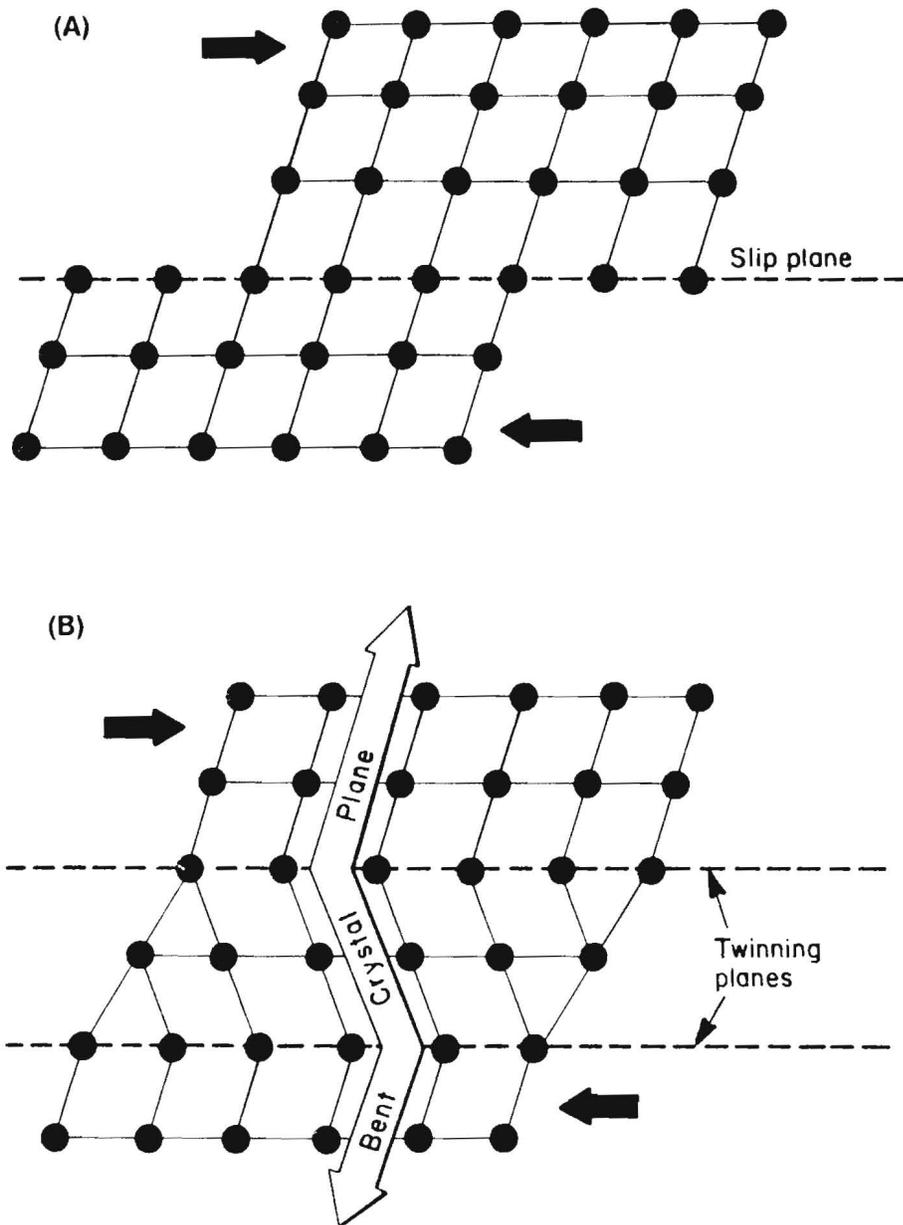
Figure 2-5. Deformation in crystal. (A) By slip.(B) By twinning. Dark arrows show direction of shearing forces causing deformation.
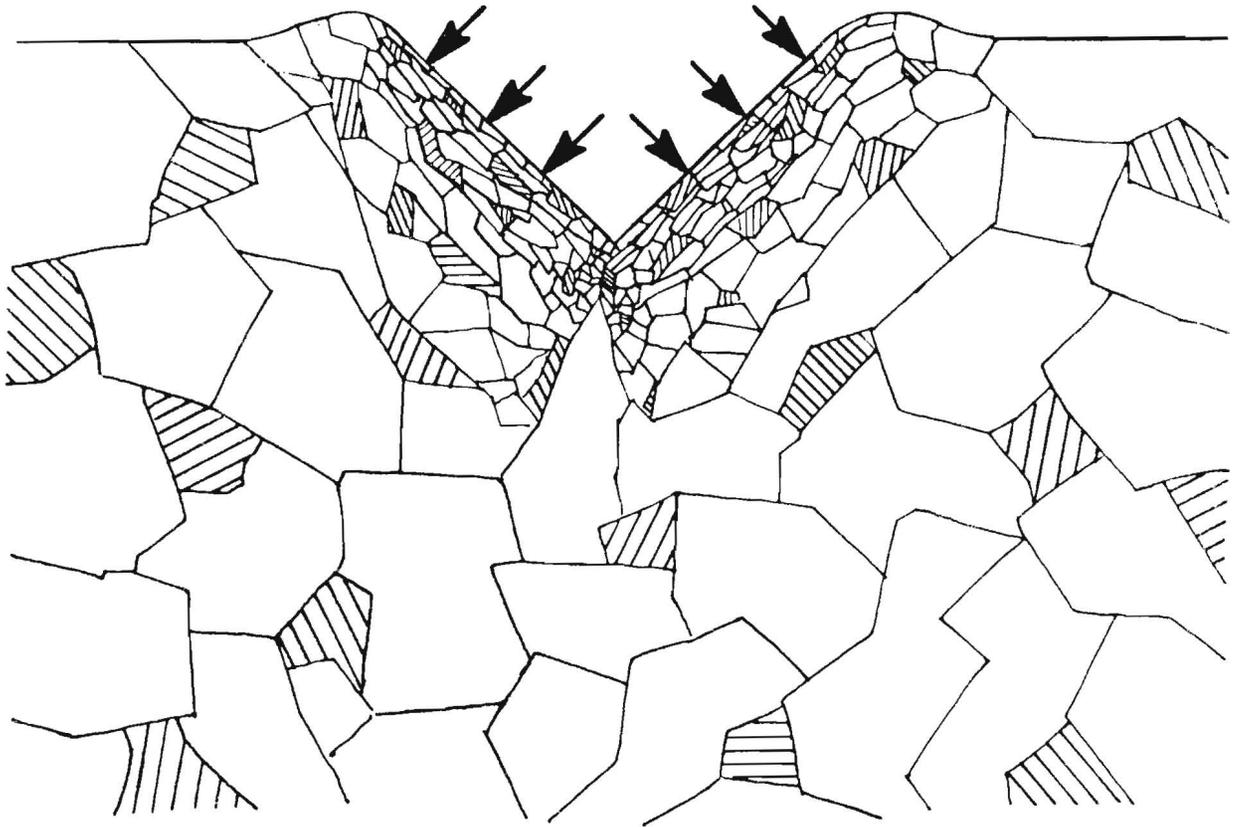
Figure 2-6. Schematic cross-sectional view of deformation in stamped number. Slip lines, twinning bands, and smaller grain size result where metal absorbs stamping compression (shown by arrows).
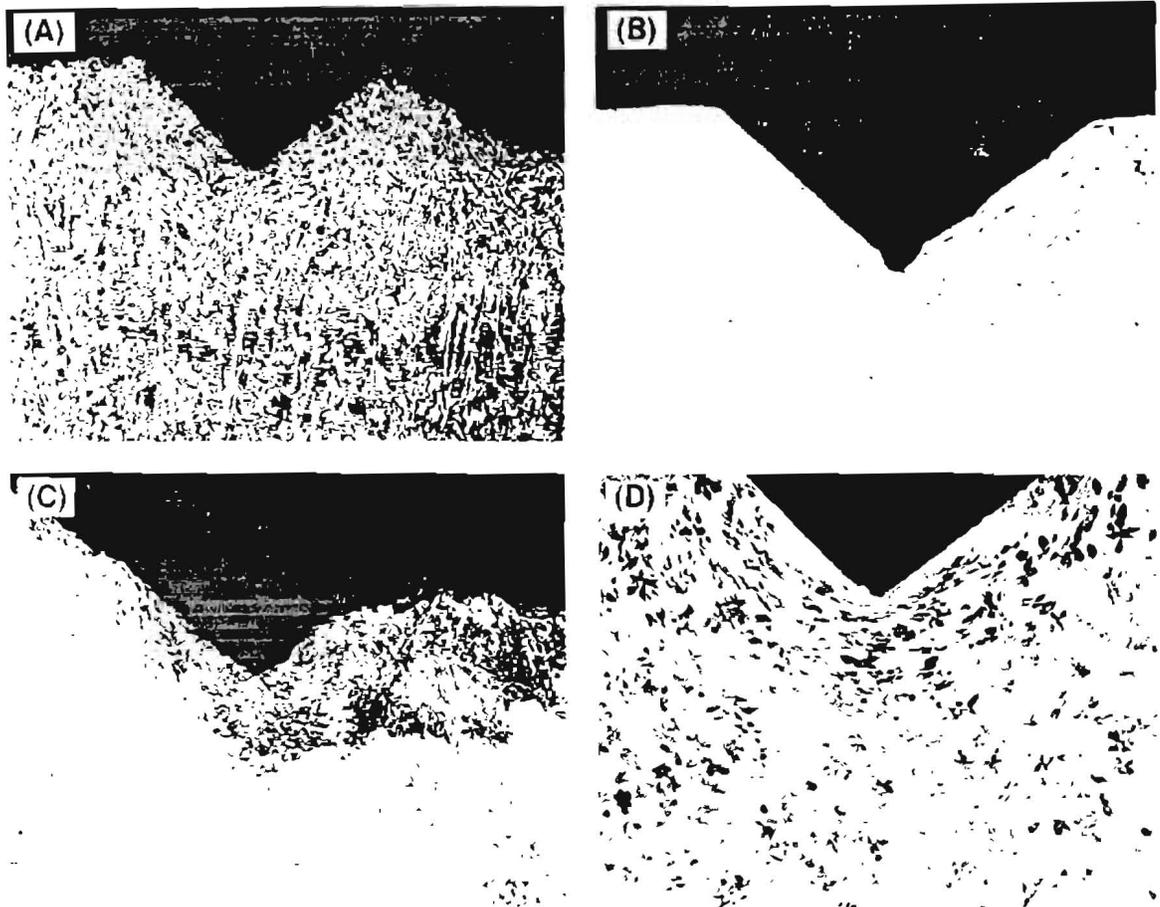
Figure 2-7. Metallographic cross sections of numerals on laboratory specimens. (A) Cast iron (50X). (B) Low-carbon steel (100X). (C) Cast brass (100X). (D) Zinc alloy (50X).

CHAPTER 3

CHEMICAL AND ELECTROLYTIC METHODS

CHEMICAL METHOD INTRODUCTION

Early in the 19th century, metallurgists began to seriously study the relationship between the microscopic structure of a metal and its mechanical properties. The importance of crystal grain size and the presence of deformation lines after cold-working became recognized. Techniques for investigating the structure of metals were refined and eventually developed into the science of metallography.

The aspect of metallography of most importance for the restoration of serial numbers is that of macroscopic examination. This is a technique which gives an overview of the gross structural features of the specimen. It consists essentially of:

(1) Surface preparation by grinding and polishing.

(2) Etching by chemical reagents to reveal structural characteristics.

(3) Examination visually or with low-power magnification.

A broad selection of macroscopic etching reagents are published in the metallurgical literature (1-5). Use of the proper reagent can reveal characteristics such as nonmetallic inclusions, porosity, segregation, cracks, depth of hardening, and fabricating defects. Each etchant is typically recommended for use only on a specific alloy. Temperature, method of application, and etching time are often prescribed.

It is not exactly certain when chemical etching techniques were first applied to the restoration of serial numbers. In the 1930's two German publications suggested the use of acid etchants for this purpose (6, 7). In the United States in 1940 a review of applications of metallurgical methods to criminal investigation was published by G. W. Pirk, a consulting metallurgist for the Utica, New York Bureau of Police. Briefly mentioned in

a footnote is the suggestion that Fry's reagent be used to recover firearm serial numbers (8). This reagent is a solution of cupric chloride and hydrochloric acid in water and has long been used by metallographers to reveal strain lines in steel. Its exact composition is listed in Table 3-1. (All reagents to be discussed in this section and as well as others selected from the criminalistic literature are listed in Tables 3-1 and 3-2.)

In 1947 Bessemans and Haemers recommended several etchants for use on specified metals (9). Particularly interesting is a solution of cupric chloride, ferric chloride, and hydrochloric acid in methyl alcohol suggested for both copper and iron alloys. A complete chapter of the 1956 English work by Nickolls is devoted to the chemical restoration method (10). Contrary to most authors, Nickolls recommends against the practice of polishing the surface prior to etching because this removes metal containing the deformation evidence. Etchants are prescribed for various steels, copper alloys, aluminum alloys, nickel, lead, gold, and platinum. Techniques for restorations on wood, plastic, leather, and painted items are also described. In 1957 Hatcher, Jury, and Weller recommended Fry's reagent for use on quality modern weapons (presumably of alloy steel) and a less reactive derivative of this reagent containing alcohol for older weapons (11).

A frequently quoted description of the chemical method appeared in the 1962 volumes by Mathews (12). The etchants recommended include several variants of Fry's reagent and also ferric chloride in water, picric acid in alcohol, dilute nitric acid, and a mixture of alcohols, nitric acid, and acetic anhydride. The tests used to evaluate the etchants are not described, but nine solutions are recommended for use on specific steels. For brass a chromic acid solution is suggested.

A unique chemical method for aluminum was proposed by Chisum in 1963 (13). The method does not rely on etching of the metal to accomplish restoration. Instead, it is based upon the rapid development of an aluminum oxide coating on the metal surface. To accomplish this the aluminum is swabbed with a solution of mercuric chloride in dilute hydrochloric acid.

The mercuric chloride chemically reacts with aluminum to produce metallic mercury, which acts as a catalyst to greatly accelerate the oxidation of aluminum by reaction with air. The selective build-up of an aluminum oxide layer renders the number visible.

The literature contains numerous additional descriptions of the chemical method. However, these works simply reiterate the recommendations of earlier authors (14-27). Thus, in spite of the large volume of literature, relatively little research has been conducted on the chemical method. The solutions appearing in the literature should not be assumed to represent a scientifically derived selection.

A recent innovation in the formulation of chemical etchants has come with the introduction of gels in place of liquids. Formulations known as Restor-A-Gel® are commercially produced for law enforcement use by Serchie Finger Print Laboratories, Moorestown, New Jersey. These paste-like gels can be applied with ease to the underside of a specimen, which can be a convenience with a bulky object, such as an engine block.

ELECTROLYTIC METHOD INTRODUCTION

In the preparation of a metal surface for microscopic examination metallographers often employ the techniques of electropolishing and electroetching (1-5). The specimen is made the anode of an electrochemical cell in which an outside dc electrical power source facilitates dissolution, or electrolysis, of the metal. In electropolishing, minute projections and irregularities on the surface are removed by this dissolution, resulting in a surface that is often of superior quality to that which can be achieved by regular polishing methods. In electroetching, the dissolution is done selectively in order to reveal desired features of the metal.

Application of the electrolytic process to accomplish serial number restorations was developed by Turner, Arai, and Mathews about 25 years ago (28, 29). In the 1953 description by Arai the specimen surface is first polished with sandpaper and cleaned with acetone. A variable dc voltage source is connected to the specimen in such a way that the specimen becomes the anode and absorbent cotton dipped in electrolytic solution becomes

the cathode of an electrochemical cell. The external voltage applied was specified by Arai to be just greater than the minimum critical voltage necessary for an electrical current to flow. For carbon steel, brass, and copper the potentials were said to be 6.0, 7.0 and 6.5 volts, respectively. Table 3-3 gives the chemical formulation for Arai's electrolytic etchant containing cupric sulfate, sulfuric acid, and gelatin in water.

The electrolytic method was described in similar terms in 1957 by Davis except for the recommendation of a different electrolytic etchant and the claim that a common 1.5-volt flashlight battery is an adequate source of electric potential (30). In more recent years various other authors have suggested additional etchants (12, 17) or have reiterated the recommendations of others (15, 18, 21, 22, 27, 31).

It is the applied electrical potential which distinguishes the electrolytic method from the more common chemical method. Investigators have generally assumed the role of this potential to be simply that of accelerating the etching process. Since chemical etchants are known which rapidly attack metals without assistance, the electrolytic method has not been extensively studied. The etchants listed in Table 3-3 from the criminal science literature should not be considered to survey the limits of the electrolytic method.

LABORATORY PROCEDURES

Laboratory specimens were stamped, obliterated, and polished as in Chapter 2. In the chemical restoration method, etchant solutions were applied to the obliterated area by use of a glass stirring rod, dropper, or cotton swab. In general, the reagent was applied to the surface and allowed in contact for 1 minute before being rinsed off with a spray of acetone. The percent of number restored was observed and recorded. If complete recovery had not been accomplished, etching treatment was continued for consecutive intervals of 2 minutes, 2 minutes, 5 minutes, and, finally, 10 minutes each, for a total of 60 minutes. Figure 3-1 shows laboratory supplies required for the chemical restoration method.

In the electrolytic method a dc power supply as in Figure 3-2 was used. The specimen was connected by wire to the positive (+) terminal. A wire from the negative (-) terminal was connected through a metal clamp to a piece of cotton dipped in the electrolytic etching solution. To accomplish restoration the obliterated area was continuously swabbed with the completely wetted cotton. Care was taken to avoid direct contact between the specimen and metal clamp. Unless otherwise stated the applied dc potential was 6 volts. The power supply was equipped with an ammeter for measuring current flow. A maximum of 1.5 amperes was allowed. Observations of the percent restored were made at regular time intervals as in the chemical method.

RESULTS AND DISCUSSION

A typical chemical restoration is illustrated in Figure 3-3. This photographic time sequence shows recovery of a number on tool steel. As time of treatment increases so does the clarity of the recovery obtained.

A survey was conducted to evaluate etchants in their ability to restore numbers. Etchants selected for testing are representative of those recommended either in the literature of criminal science (6-27) or metallography (1-5). The five ferrous and three nonferrous metals described in Chapter 2 were investigated. Testing results for these two metal classifications are given in Tables 3-4 and 3-5. The tables list the maximum percent of a just-obliterated number recovered and the amount of etching time required.

Results on iron and steel (Table 3-4) show that complete recovery of a just-obliterated number can be accomplished in a short time with a wide variety of etchants. A few general observations appear:

(1) The time required to accomplish a restoration follows that expected from the chemical reactivities of the etchants. For example, 25% nitric acid works more rapidly than either 1% nitric acid or 10% nitric acid in ethyl alcohol.

(2) The time required is also a function of the metal. The order of chemical reactivity is seen to be:

low-carbon steel > alloy steel = tool steel >

cast iron > stainless steel

An exception to this order is the rapid restoration
attained on stainless steel by the three etchants con-
taining cupric compounds and hydrochloric acid.

(3) Etchants performing the best with respect to clarity and
rapidity are Fry's reagent, acidic cupric sulfate, am-
monium persulfate, and 25% nitric acid.

Results on aluminum alloy, brass, and zinc alloy (Table 3-5) likewise
show that complete recovery of just-obliterated numbers can be easily ac-
complished on nonferrous metals.

Aluminum is one of the more reactive metals with respect to attack
from chemical solutions. The reagents judged best on aluminum alloy are
cupric chloride in nitric acid, acidic ferric chloride, ferric chloride,
acidic mercuric chloride, and hydrofluoric acid in mixed acids. The third
of these is unique in that it is not an etchant. It is the reagent recommended
by Chisum exclusively for aluminum (13). As described previously, its
role is to catalyze the selective growth of aluminum oxide on the metal
surface. In accordance with the procedure of Chisum, the aluminum alloy
was cleaned with dilute sodium hydroxide prior to application of the acidic
mercuric chloride. After removal of this reagent the oxide coating rapidly
formed, but less so immediately over the obliterated number.

Brass is a comparatively inert metal, but it also undergoes deforma-
tion readily when cold-worked (see Figure 2-4). Complete restorations
were attained with several etchants, the best of which were judged to be
acidic ferric chloride, chromic acid, hydrogen peroxide, and 25% nitric
acid. Hydrogen peroxide is not a typical etchant; upon contact with brass
it liberates oxygen gas and covers the brass with a black coating of cupric
oxide.

Zinc alloy is the most reactive of the metals in this survey with respect
to attack by acids. Complete restorations were frequently attained in 1 min-
ute or less. Etchants considered preferable by these results are ferric
chloride, chromic acid, iodine, and 5% hydrochloric acid in ethyl alcohol.

The technique of alternate treatments of 50% hydrochloric acid and 50% nitric acid (27) was judged overly reactive.

Figure 3-4 shows the variety in appearance of numbers recovered by the chemical method on alloy steel, stainless steel, aluminum alloy, and brass.

A survey of the electrolytic method was conducted in a similar fashion. Results obtained on the five ferrous and three nonferrous metals are given in Tables 3-6 and 3-7, respectively. In addition to listing the maximum percent of a just-obliterated number recovered and the time required, these tables list the current flow in amperes during electrolysis.

In cases where direct comparison can be made of a specific metal and etchant, the electrolytic method is seen to require less time than the chemical method. Taking the data as a whole, however, the electrolytic method appears not to be faster, because the etchants chosen for it tended to be inherently less chemically reactive than for the chemical method.

The electrolytic etchants which performed best with respect to clarity and rapidity for the metals of Tables 3-6 and 3-7 are as follows:

Iron and steel:	Davis' reagent
	Turner's reagent
	ammonium persulfate
	10% hydrochloric acid in
	methyl alcohol
Aluminum alloy:	ferric chloride
	hydrofluoric acid in glycerol
	hydrofluoric acid in mixed acids
	10% sodium hydroxide
Brass:	acidic ferric chloride
	25% nitric acid
Zinc alloy:	chromic acid
	10% sodium hydroxide

Figure 3-5 shows electrolytic restorations on cast iron and stainless steel. They are similar in appearance to chemical restorations.

THEORY

The chemical and electrolytic restoration methods are of interest from a theoretical viewpoint. A simplified description is given here of the scientific principles upon which these methods rest.

When a metal is etched, its surface is chemically dissolved by the process known as oxidation. Atoms of metal become oxidized by losing one or more of their electrons and, thereby, are transformed into positively charged ions. In this new form they are soluble in the etchant solution. The process can be illustrated by the dissolution of zinc by hydrochloric acid:

$$Zn + 2HCl \longrightarrow ZnCl_2 + H_2$$

Metallic zinc is converted to zinc ion, in the form of the soluble compound, zinc chloride. Hydrogen gas is a second product of the reaction. Because of the role it plays, hydrochloric acid is designated the oxidizing agent. In the reaction, hydrochloric acid is reduced while zinc is oxidized.

In the electrolytic method, etching occurs in the same fashion except that the outside electrical power source acts as a sort of electron "pump". Using the example above, the zinc metal becomes an anode and undergoes oxidation. The oxidation half-reaction represented:

$$Zn \longrightarrow Zn^{2+} + 2e^-$$
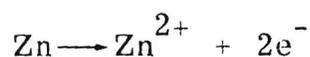
The electrons that are liberated pass through the external wire and power source to the cotton-containing metal clamp. At this cathode, hydrogen ion is reduced according to the half-reaction:

$$2H^+ + 2e^- \longrightarrow H_2$$
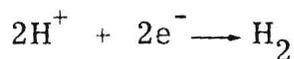
Thus, the overall reaction is identical to that shown above, since zinc ion and hydrogen gas are the two products in both cases. Distinctions of the electrolytic method are the separate physical locations of the two half-reactions and the fact that a given reaction will occur faster with the aid of the power source.

Metals vary in their ability to lose electrons and can be ranked according to tendency to become oxidized. The resulting ranking is known in chemistry as the electromotive series. A shortened version of the electromotive series is shown in Table 3-8. The standard oxidation potential listed for each metal is a measure of its ten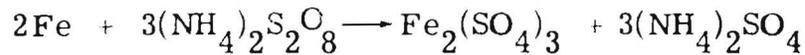dency to lose electrons under certain standardized conditions. The unit used for the scale is the volt, and hydrogen is assigned a value of zero by convention. A high oxidation potential, as for sodium, implies the metal will react vigorously with even the weakest of oxidizing agents. On the other extreme, gold has a very low oxidation potential and is quite unreactive. Caution must be exercised in drawing conclusions about practical situations from the electromotive series. For example, aluminum and chromium are slow to oxidize under some conditions. This passivity results from a protective layer of metal oxide which readily forms on the surface. Similarly, when alloying elements are added to a metal, its chemical reactivity is altered. The inertness of stainless steel illustrates the extent to which alteration can occur. (Recall that the ferrous metals of this study displayed varying susceptibility to attack from etchants.)

Oxidizing agents can similarly be ranked according to their tendency for reaction. Those found in the chemical and electrolytic etchants of this study are listed in Table 3-9. Since oxidizing agents undergo reduction, the value given for each one is its standard reduction potential. A high value indicates a strong oxidizing agent, as for ammonium persulfate or hydrogen peroxide. In contrast to these reagents, hydrogen ion from hydrochloric acid — at the bottom of Table 3-9 — is not usually referred to as an oxidizing agent at all, although it plays this role with reactive metals.
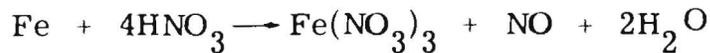
In general, rapid chemical etching will result when the combined factors of metal oxidation potential and etchant reduction potential are sufficiently high. For example, Fry's reagent reacts readily with all ferrous metals. The principal chemical reactions occurring are:

$$Fe + 2HCl \longrightarrow FeCl_2 + H_2$$

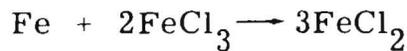$$Fe + CuCl_2 \longrightarrow FeCl_2 + Cu$$

Under suitable conditions both hydrogen gas and copper metal can be observed as products. Analogous reactions occur with even greater vigor for aluminum and zinc. On the other hand, brass reacts more slowly. To further illustrate the reactions between etchants and metals, the following equations show the action on ferrous metals of ammonium persulfate:

$$2Fe + 3(NH_4)_2S_2O_8 \longrightarrow Fe_2(SO_4)_3 + 3(NH_4)_2SO_4$$

dilute nitric acid:

$$Fe + 4HNO_3 \longrightarrow Fe(NO_3)_3 + NO + 2H_2O$$

and ferric chloride:

$$Fe + 2FeCl_3 \longrightarrow 3FeCl_2$$

Aluminum has some unique reactions by virtue of its high position in the electromotive series. As previously described, Chisum's method (13) is applicable only to this metal. The first reaction that occurs is:

$$2Al + 3HgCl_2 \longrightarrow 2AlCl_3 + 3Hg$$

This process deposits the trace amounts of mercury required to catalyze aluminum oxidation by air:

$$4Al + 3O_2 \longrightarrow 2Al_2O_3$$
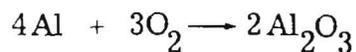
Aluminum is also unique in its reactivity to alkalies. Thus, 10% sodium hydroxide is an etchant for this metal by the reaction:

$$2Al + 2NaOH + 2H_2O \longrightarrow 2AlO_2^- + 2Na^+ + 3H_2$$

Prior to the oxidation step shown, the sodium hydroxide serves to dissolve the protective oxide coating on the metal.

To be successful for restoration purposes an etchant must do more than attack a metal rapidly. It must attack selectively so that the number becomes visible against its surroundings. As discussed in Chapter 2, the rate of chemical etching of a metal is increased by cold-working. The chemical restoration on tool steel illustrated in Figure 3-3 clearly occurrs because the deformed metal immediately below the number is etched more rapidly than its surroundings.

In the chemical and electrolytic restorations of Figure 3-4 and 3-5, numbers are restored not because they are <u>observably</u> etched more rapidly but because etching makes them appear lighter or darker than their surroundings. Restorations of this nature can also be attributed to more rapid etching of deformed metal. Such etching causes the metal that forms the image of the number to differ in reflecting ability from its background. For some metals the number has a well-defined border enhancing its visibility. As illustrated in Figure 2-6, compression that occurs during stamping has a considerable horizontal component. For this reason, it is possible for metal on both sides of the stamped groove to contain more deformation or deformation of a different kind than metal immediately below the groove.

The chemical restoration on aluminum alloy (Figure 3-4) is of interest because the etchant has revealed individual metal grains. The number is visible because its grains are less clearly defined. A different restoration mechanism may be operative here. This mechanism is based upon the fact that twinning defects will be more common in the deformed metal below the number. Figure 3-6 illustrates the effect a twin can have on the light reflectance of a crystal grain. When the grain has received only polishing, there is no effect. But if polishing is followed by etching, the grain surface reflects light diffusely at the twin. The result will be a different appearance for grains having twins. Note that this restoration mechanism does not depend upon a difference in dissolution rates of deformed and nondeformed metal.

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Table 3-1

Chemical Etchants for Iron and Steel

Selected from the Criminal Science Literature

Literature references are indicated in parentheses. See end of chapter for listings.

Solutions of cupric compounds in acid

1. Fry's reagent
90 g $CuCl_2$
120 ml HCl
100 ml water
for rolled and cast
steel (8-12, 17, 18, 24)

2. 8 g $CuCl_2$
16 g $NiCl_2$
140 ml HCl
50 ml water (23)

3. 5 g $CuCl_2$
40 ml HCl
25 ml ethyl alcohol
30 ml water
for cast steel (11, 12, 17
18, 20, 22)

4. 6 g $CuCl_2$
8 g $FeCl_3$
12 ml HCl
100 ml methyl alcohol
(9, 15)

Solutions of other inorganic compounds

1. Ferric chloride in acid
5 g $FeCl_3$
50 ml HCl
100 ml water
for stainless and high-speed
steel (17)

2. Ferric chloride
6% $FeCl_3$ in water
for rolled and cast steel
(12, 17, 18)

3. Mercuric nitrate in acid
7 parts $Hg(NO_3)_2$
100 parts HCl
100 parts water (25)

4. Ammonium persulfate
10% $(NH_4)_2S_2O_8$ in water
for rolled and malleable
cast steel and cast iron
(12, 17, 18)

Solutions of mineral or organic acids

1. Chromic acid
40 g CrO_3
50 ml water (23)

2. 50% hydrochloric acid
100 parts HCl
100 parts water
for stainless steel (25)

5. Potassium dichromate
$K_2Cr_2O_7$ in 10% sulfuric
acid for cast steel or
cast iron (10, 14)

6. Aqua regia
75 ml HCl
25 ml HNO_3
for stainless steel and
high-speed steel (17)

Table 3-1 (Cont.)

3. Picric acid saturated in ethyl alcohol (12, 17, 18)

7. Picric acid in acidic ethyl alcohol
    1 g picric acid
    2 drops HCl
    25 ml ethyl alcohol (25)

4. 4% nitric acid in amyl alcohol (12, 17, 18)

8. 1% HNO_3 in water (12, 17, 18)

Table 3-2

Chemical Etchants for Nonferrous Metals

Selected from the Criminal Science Literature

Literature references are indicated in parentheses. See end of chapter for listings.

Aluminum and its alloys

1. Hume-Rothery's solution
   200 g $CuCl_2$
   5 ml HCl
   100 ml water (10, 14)

2. 25% nitric acid
   25 ml HNO_3
   75 ml water (17, 18)

3. Nitric and phosphoric acids
   6 ml HNO_3
   94 ml H_3PO_4 (17)

4. Villella's solution
   2 parts HF
   1 part HNO_3
   3-4 parts glycerol
   (10, 14, 17, 18)

5. Ferric chloride
   6% $FeCl_3$ in water (26)

6. Phosphoric acid
   15% or 25% H_3PO_4
   in water (25)

7. Mixed acids
   1.0 ml HF
   1.5 ml HCl
   2.5 ml HNO_3
   95.0 ml H_2O (25)

8. 10% NaOH and 10% HNO_3,
   used in alternate treat-
   ments (24, 25)

9. 1% NaOH solution (17, 18)

Brass and copper

1. 40 g $CuCl_2$
   180 ml HCl
   100 ml water (10)

2. 6 g $CuCl_2$
   8 g $FeCl_3$
   12 ml HCl
   100 ml methyl alcohol
   (19)

3. 20 g CrO_3
   1.5 g Na_2SO_4
   100 ml water (12, 17, 18)

5. 1.5 g $CuCl_2$
   30 ml HCl
   30 ml ethyl alcohol
   95 ml water (25)

6. 19 g $FeCl_3$
   6 ml HCl
   100 ml water (10, 14)

7. 20 g CrO_3
   50 ml HNO_3
   30 ml water (25)

Table 3-2 (Cont.)

4. Nitric acid of varied
    concentration (17, 18)

Chromium and nickel

Mixed acids in glycerine
1 part HNO_3
3 parts HCl
2 parts glycerol (25)

Lead

1. Nitric acid, concentrated (10)

2. Hydrogen peroxide in acid
    1 part 10% H_2O_2
    3 parts acetic acid (25)

3. 5% $AgNO_3$ (10)

4. Molybdic acid
    100 g H_2MoO_4
    60 ml HNO_3
    140 ml NH_4OH
    240 ml water (17, 21)

Magnesium and its alloys

10% Malic acid (24)

Precious metals

1. Bromine water, for
    gold and platinum (10)

2. Mixed acids
    1 part HNO_3
    1 part HCl
    6 parts water
    for silver (25)

3. Aqua regia
    75 ml HCl
    25 ml HNO_3
    for gold and platinum (21)

4. Nitric acid, dilute,
    for silver (21)

Tin

Hydrogen peroxide in acid
1 drop H_2O_2
50 ml acetic acid
50 ml water (24, 25)

Zinc and its alloys

1. Hydrochloric acid,
    diluted as necessary (25)

2. 50% HCl and 50% HNO_3,
    used in alternate treat-
    ments (25)

Table 3-3

Electrolytic Etchants
Selected from the Criminal Science Literature

Literature references are indicated in parentheses. See end of chapter
for listings.

1. Cupric sulfate
   1 g $CuSO_4$
   15 ml H_2SO_4
   1 g   gelatin
   500 ml water
   for general use (12, 15, 29)

2. Cupric chloride
   2.5 g $CuCl_2$
   40 ml   HCl
   25 ml   ethyl alcohol
   30 ml   water
   for rolled steel, cast
   iron, brass (12)

3. 2% fluoroboric acid, for
   aluminum (17)

4. Cupric ammonium chloride
   5 g $CuCl_2 \cdot 2NH_4Cl \cdot 2H_2O$
   50 ml HCl
   50 ml water
   for steel (30)

5. 17% sulfuric acid, for
   aluminum (17)

6. Chromic acid
   20 g CrO_3
   1.5 g Na_2SO_4
   100 ml water (12)

Table 3-4

Chemical Method Restoration Results on Iron and Steel

Chemical etchant	Percent recovered and time required for just-obliterated specimen				
	Alloy steel	Cast iron	Low-carbon steel	Tool steel	Stainless steel
Fry's reagent 90 g $CuCl_2$ 120 ml HCl 100 ml water	80% (1 min)	100% (1 min)	100% (<1 min)	100% (1 min)	100% (1 min)
Acidic cupric sulfate 20 g $CuSO_4$ 100 ml HCl 100 ml water	100% (1 min)	100% (20 min)	100% (<1 min)	100% (3 min)	100% (1 min)
Acidic cupric chloride 5 g $CuCl_2$ 60 ml HCl 60 ml water	70% (10 min)	0% (60 min)	100% (<1 min)	100% (1 min)	100% (1 min)
Acidic ferric chloride saturated $FeCl_3$ in HCl	100% (1 min)	100% (60 min)	100% (<1 min)	30% (5 min)	100% (30 min)
Ferric chloride 6 g $FeCl_3$ 93 ml water	100% (10 min)	20% (3 min)	100% (2 min)	100% (3 min)	0% (60 min)
Amonium persulfate 10 g $(NH_4)_2S_2O_8$ 90 ml water	100% (1 min)	100% (3 min)	100% (<1 min)	100% (3 min)	0% (60 min)
25% nitric acid 25 ml HNO_3 75 ml water	100% (1 min)	100% (3 min)	100% (<1 min)	100% (1 min)	0% (60 min)

Table 3-4 (Cont.)

Chemical etchant	Percent recovered and time required for just-obliterated specimen				
	Alloy steel	Cast Iron	Low-carbon steel	Tool steel	Stainless steel
1% nitric acid __1 ml HNO₃__ 99 ml water	100% (1 min)	100% (60 min)	100% (<1 min)	100% (3 min)	0% (60 min)
Mixed acids __38 ml HCl__ 12 ml H₂SO₄ 50 ml water	100% (30 min)	100% (15 min)	100% (10 min)	100% (10 min)	0% (60 min)
10% nitric acid in ethyl alcohol __10 ml HNO₃__ 90 ml ethyl alcohol	95% (1 min)	100% (30 min)	100% (<1 min)	100% (3 min)	0% (60 min)
10% hydrochloric acid in methyl alcohol __10 ml HCl__ 90 ml methyl alcohol	50% (60 min)	100% (30 min)	100% (2 min)	100% (30 min)	100% (30 min)

Table 3-5

Chemical Method Restoration Results on Nonferrous Metals

	Chemical etchant	Percent recovered of just-obliterated specimen	Time required
Aluminum alloy	Fry's reagent 90 g $CuCl_2$ 120 ml HC 100 ml water	85%	10 min
	Cupric chloride in nitric acid 5 g $CuCl_2$ 3 ml HNO_3 100 ml water	100%	3 min
	Acidic ferric chloride 25 g $FeCl_3$ 25 ml HCl 100 ml water	100%	5 min
	Ferric chloride 25 g $FeCl_3$ 100 ml water	100%	5 min
	Acidic mercuric chloride 13.6 g $HgCl_2$ 4.0 ml HC1 500 ml water	100%	1 min
	Nitric acid concentrated	50%	45 min
	25% nitric acid 25 ml HNO_3 75 ml water	30%	45 min

Table 3-5 (Cont.)

	Chemical etchant	Percent recovered of just-obliterated specimen	Time required
Aluminum alloy (continued)	Acids in glycerol 40 ml HCl 10 ml HNO$_3$ 50 ml glycerol	70%	45 min
	Hydrofluoric acid in mixed acids 2 ml HF 24 ml HCl 12 ml HNO$_3$ 2 ml water	100%	1 min
	10% sodium hydroxide 10 g NaOH 90 ml water	70%	40 min
	1% sodium hydroxide 1 g NaOH 99 ml water	60%	40 min
Brass	Acidic cupric chloride 30 g CuCl$_2$ 40 ml HCl 30 ml water	95%	10 min
	Acidic ferric chloride 25 g FeCl$_3$ 25 ml HCl 100 ml water	100%	1 min
	Chromic acid 20 g CrO$_3$ 1.5 g Na$_2$SO$_4$ 100 ml water	100%	5 min
	Ammonium persulfate 10 g (NH$_4$)$_2$S$_2$O$_8$ 90 ml water	40%	45 min
	Hydrogen peroxide 20 ml H$_2$O$_2$, 30% 50 ml NH$_4$OH 50 ml water	100%	10 min

Table 3-5 (Cont.)

	Chemical etchant	Percent recovered of just-obliterated specimen	Time required
Brass (continued)	25% nitric acid 25 ml HNO_3 75 ml water	100%	5 min
	6% nitric acid 6 ml HNO_3 94 ml water	15%	45 min
	Ammonium hydroxide concentrated	60%	45 min
Zinc alloy	Fry's reagent 90 g $CuCl_2$ 120 ml HCl 100 ml water	90%	5 min
	Ferric chloride 25 g $FeCl_3$ 100 ml water	100%	10 min
	Chromic acid 20 g CrO_3 1.5 g Na_2SO_4 100 ml water	100%	1 min
	Iodine 10 g I_2 30 g KI 100 ml water	100%	1 min
	50% hydrochloric acid and 50% nitric acid alternate treatments	100%	1 min
	15% nitric acid in methyl alcohol 15 ml HNO_3 85 ml methyl alcohol	100%	1 min
	5% hydrochloric acid in ethyl alcohol 5 ml HCl 95 ml ethyl alcohol	100%	1 min

Table 3-5 (Cont.)

	Chemical etchant	Percent recovered of just-obliterated specimen	Time required
Zinc alloy (continued)	10% sodium hydroxide 10 g NaOH 90 ml water	0%	60 min

Table 3-6

Electrolytic Method Restoration Results on Iron and Steel

Electrolytic etchant	Percent recovered, time required, and current for just-obliterated specimen[a]				
	Alloy steel	Cast iron	Low-carbon steel	Tool steel	Stainless steel
Davis' reagent (modified) 5 g $CuCl_2$ 50 ml HCl 50 ml H_2O	100% (10 sec) 1.5 A	100% (1 min) 1.5A	100% (2 sec) 1.5A	100% (1 min) 1.5A	100% (1 min) 1.4A
Turner's reagent 2.5 g $CuCl_2$ 40 ml HCl 25 ml ethyl alcohol 30 ml water	100% (10 sec) 1.5 A	100% (3 min) 1.5A	100% (2 sec) 1.5A	100% (1 min) 1.5A	100% (1 min) 1.4A
Arai's reagent 1 g $CuSO_4$ 15 ml H_2SO_4 1 g gelatin 500 ml water	100% (10 sec) 1.1 A	100% (1 min) 1.5 A	100% (4 min) 1.1 A	100% (1 min) 1.5 A	100% (1 min) 1.2 A
Sodium carbonate 10 g Na_2CO_3 90 ml water	0% (10 min) 0.6 A	0% (20 min) 0.8 A	100% (15 min) 1.4 A	0% (20 min) 0.6 A	0% (30 min) 0.6 A
Chromic acid 10 g CrO_3 90 ml water	80% (1.5 min) 1.4 A	100% (15 min) 1.5 A	100% (1 min) 1.5 A	100% (1 min) 1.5 A	100% (3 min) 1.4 A
Ammonium persulfate 10 g $(NH_4)_2S_2O_8$ 90 ml water	100% (10 sec) 1.4 A	100% (1 min) 1.5 A	100% (15 sec) 1.5 A	100% (1 min) 1.3 A	100% (3 min) 1.2 A

Table 3-6 (Cont.)

Electrolytic etchant	Percent recovered, time required, and current for just-obliterated specimen[a]				
	Alloy steel	Cast iron	Low-carbon steel	Tool steel	Stainless steel
10% hydrochloric acid in methyl alcohol 10 ml HCl 90 ml methyl alcohol	100% (30 sec) 0.4 A	100% (5 min) 0.5 A	100% (30 sec) 0.2 A	100% (1 min) 0.7 A	100% (3 min) 0.4 A
10% oxalic acid 10 g oxalic acid 90 ml water	100% (45 sec) 0.3 A	100% (5 min) 0.9 A	0% (20 min) 1.1 A	25% (1 min) 0.7 A	100% (5 min) 0.6 A
0.5% sodium hydroxide 0.5 g NaOH 99.5 ml water	0% (10 min) 0.1 A	0% (20 min) 0.3 A	0% (20 min) 0.4 A	0% 20 min) 0.3 A	0% (30 min) 0.2 A

[a] Applied dc potential is 6.0 volts maximum throughout; maximum current is 1.5 A.

Table 3-7

Electrolytic Method Restoration Results on Nonferrous Metals

	Electrolytic etchant	Percent recovered for just-obliterated specimen	Time required	Current[a]
Aluminum alloy	Arai's reagent 1 g $CuSO_4$ 15 ml H_2SO_4 1 g gelatin 500 ml water	0%	30 min	0.1 A
	Ferric chloride 25 g $FeCl_3$ 100 ml water	100%	3 min	0.9 A
	25% nitric acid 25 ml HNO_3 75 ml water	85%	30 min	1.5 A
	10% sulfuric acid 10 ml H_2SO_4 90 ml water	0%	30 min	0.1 A
	Hydrofluoric acid in glycerol 10 ml HF 55 ml glycerol 35 ml water	100%	5 min	0.1 A
	Hydrofluoric acid in mixed acids 2 ml HF 24 ml HCl 12 ml HNO_3 2 ml water	100%	<1 min	1.5 A
	10% sodium hydroxide 10 g NaOH 90 ml water	100%	10 min	1.5 A
	1% sodium hydroxide 1 g NaOH 99 ml water	100%	15 min	0.1 A

Table 3-7 (Cont.)

	Electrolytic etchant	Percent recovered for just-obliterated specimen	Time required	Current[a]
Brass	Acidic ferric chloride 25 g $FeCl_3$ 25 ml HCl 100 ml water	100%	10 sec	1.5 A
	Chromic acid 20 g CrO_3 1.5 g Na_2SO_4 100 ml water	100%	1 min	1.5 A
	25% nitric acid 25 ml HNO_3 75 ml water	100%	1 min	1.5 A
	30% phosphoric acid 30 ml H_3PO_4 70 ml water	100%	5 min	1.5 A
Zinc alloy	Chromic acid 20 g CrO_3 1.5 g Na_2SO_4 100 ml water	100%	5 sec	1.3 A
	10% sodium hydroxide 10 g NaOH 90 ml water	100%	1 min	1.3 A

[a] Applied dc potential is 6.0 volts maximum throughout; maximum current allowed is 1.5 A.

Table 3-8

Electromotive Series for Metals
of Interest in the Present Study

Metal	Metallic form/Oxidized form	Standard oxidation potential, volts
Sodium	Na/Na^{+}	+2.71
Magnesium	Mg/Mg^{2+}	+2.36
Aluminum	Al/Al^{3+}	+1.66
Zinc	Zn/Zn^{2+}	+0.76
Chromium	Cr/Cr^{3+}	+0.74
Iron	Fe/Fe^{2+}	+0.44
Nickel	Ni/Ni^{2+}	+0.25
Tin	Sn/Sn^{2+}	+0.14
Lead	Pb/Pb^{2+}	+0.13
Hydrogen	H_2/H^{+}	0.00
Copper	Cu/Cu^{2+}	-0.34
Silver	Ag/Ag^{+}	-0.80
Gold	Au/Au^{3+}	-1.50

Table 3-9

Oxidizing Agents of Interest in the Present Study

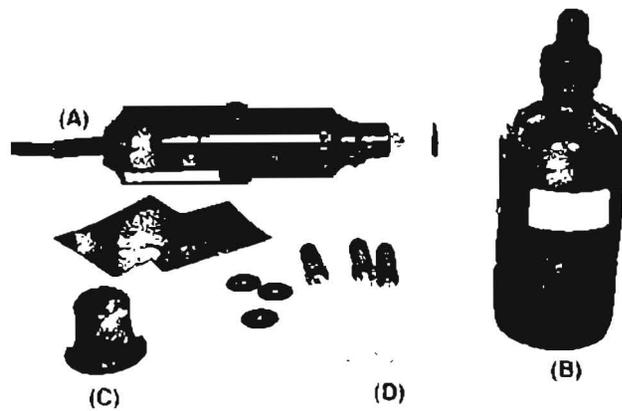| Reagent used in etchant formulations	Oxidizing agent/Reduced form	Standard reduction potential, volts
$(NH_4)_2S_2O_8$	$S_2O_8^{2-}/SO_4^{2-}$	+2.01
H_2O_2	H_2O_2/H_2O	+1.78
CrO_3 in acid	$Cr_2O_7^{2-}/Cr^{3+}$	+1.33
O_2 in air	O_2/H_2O	+1.19
HNO_3 (dilute)	NO_3^-/NO	+0.96
HNO_3 (conc.)	NO_3^-/HNO_2	+0.94
$HgCl_2$	Hg^{2+}/Hg	+0.85
$FeCl_3$	Fe^{3+}/Fe^{2+}	+0.77
I_2 in KI solution	I_3^-/I^-	+0.54
$CuCl_2$ and $CuSO_4$	Cu^{2+}/Cu	+0.34
HCl	H^+/H_2	0.00

Figure 3-1. Specimen polishing and chemical restoration method supplies.
(A) Power tool for polishing and fine grinding. (B) Chemical etchant.
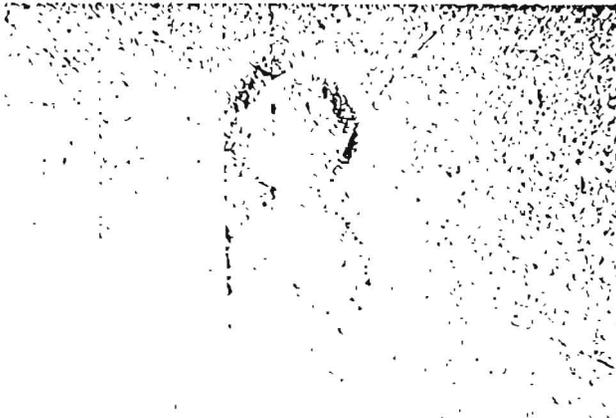(C) Jeweler's eyepiece. (D) Applicators.

Figure 3-2. Electrolytic restoration method apparatus. Dc power supply connected to pistol specimen and cotton swab.
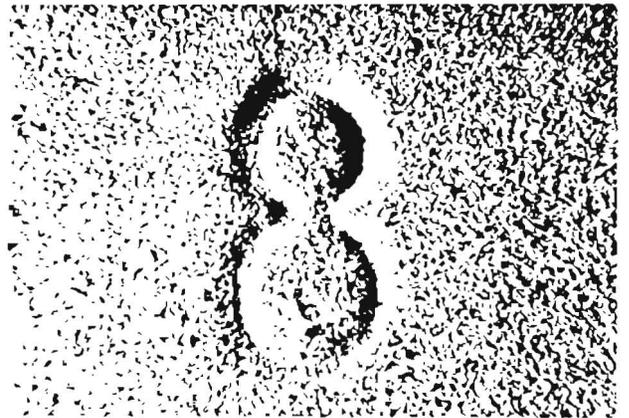
0 minutes

15 minutes

30 minutes

120 minutes

Figure 3-3. Restoration sequence by chemical method on just-obliterated tool steel (8X). Total time of etching is indicated for each figure; chemical etchant is Fry's reagent.
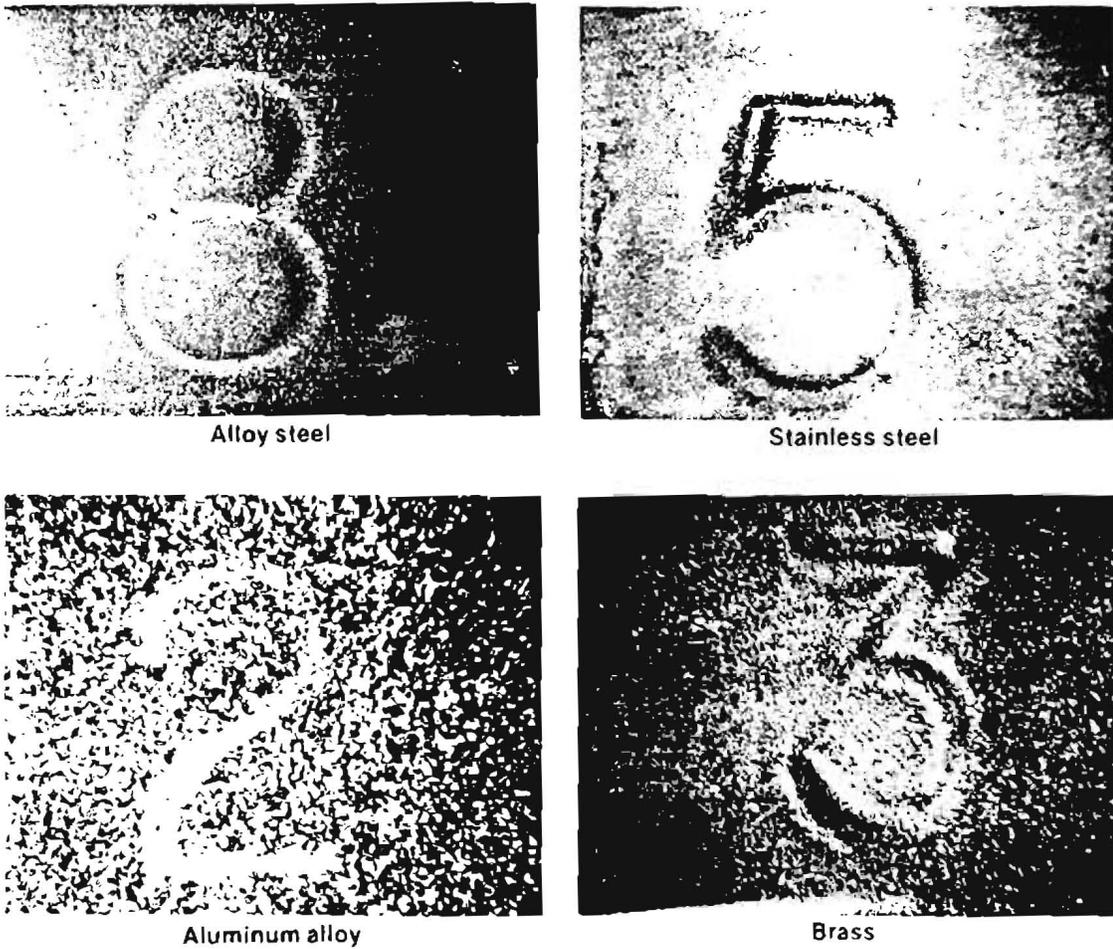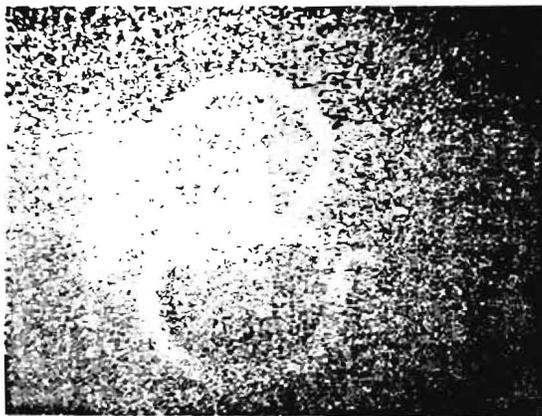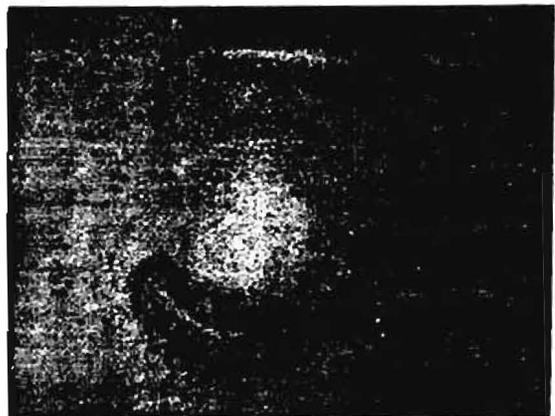
Figure 3-4. Chemical method restorations on selected metals (8X).
Etchants used are: ammonium persulfate on alloy steel, Fry's reagent
on stainless steel, hydrofluoric acid in mixed acids on aluminum alloy,
and 25% nitric acid on brass. (See Tables 3-4 and 3-5 for etchant
formulations.)

Cast iron   Stainless steel

Figure 3-5.   Electrolytic method restorations on cast iron and stainless steel (8X).   Electrolytic etchants used are Arai's and Davis' reagent, respectively.   (See Table 3-6 for etchant formulations.)
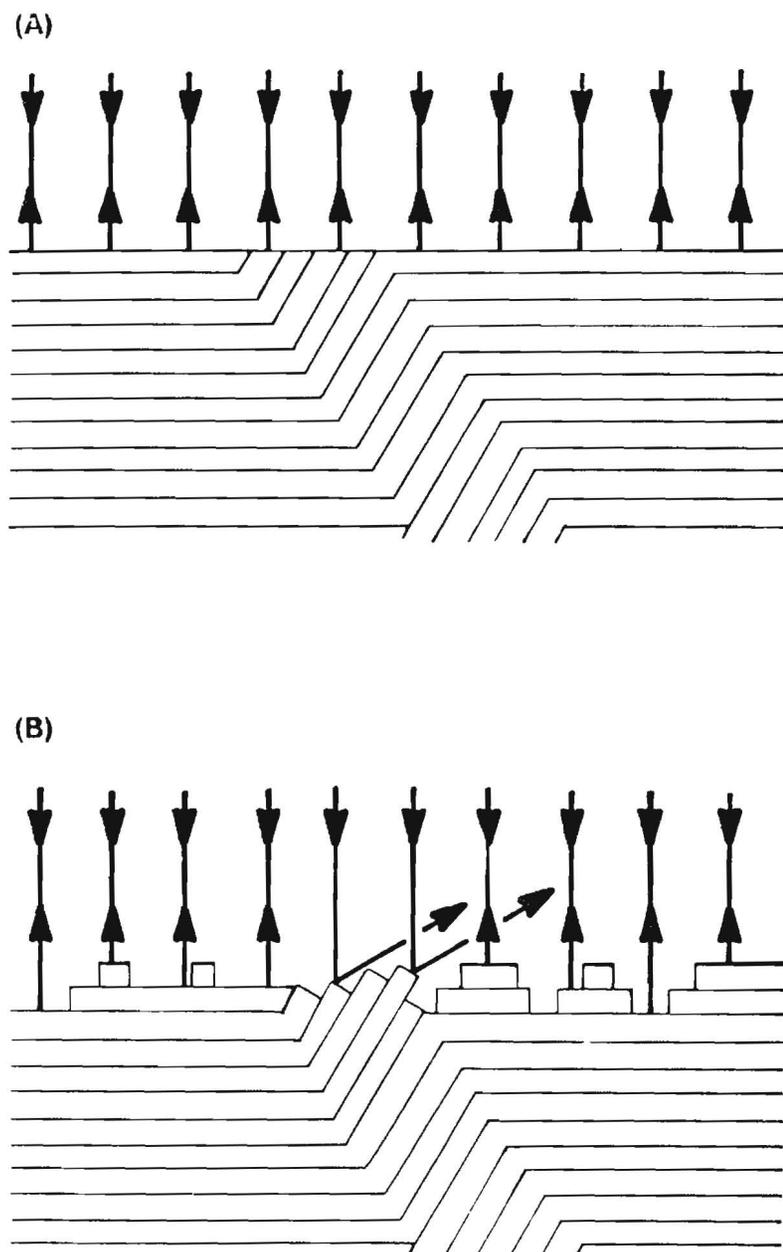
Figure 3-6. Light-reflecting properties of metal grain with twinning defect. (A) After polishing. (B) After polishing and etching.

CHAPTER 4.

ULTRASONIC CAVITATION ETCHING METHOD

INTRODUCTION

Cavitation is a phenomenon that can be made to occur in a liquid. It is the formation of vapor bubbles due to localized reductions in pressure. Thus, the phenomenon is similar to boiling, which is the formation of bubbles by localized increases in temperature. A solid object moving rapidly through a liquid can create the reduction in pressure necessary for cavitation. This can be seen when a motor boat propeller spinning in water creates a stream of bubbles that rise to the surface.

To hydraulic engineers cavitation is a troublesome phenomenon. It causes damage to pumps, ship propellers, and pipes carrying liquids. The bubbles are of high energy and have the effect of etching the metal surface from which they emerge. Because of these practical consequences, the subject of cavitation damage has been widely studied. A comprehensive review has recently been written (1). One specific area of concern over the effects of cavitation damage is in the design of fuel lines for space exploration vehicles. For this reason National Aeronautics and Sapce Administration engineers have extensively investigated cavitation induced in liquids by high-frequency vibrations (2, 3).

In 1972, as part of a program to find new applications for space technology, S.G. Young of NASA Lewis Research Center tested the feasibility of using cavitation etching to restore serial numbers. The apparatus used by NASA to induce cavitation in water is similar to that shown in Figure 4-1. It consists of a power supply that converts 60-Hz alternating electrical current into 20-kHz electrical current. This current is delivered to a converter, where a piezoelectric transducer transforms the energy into mechanical vibrations of the same frequency. The vibrations are transmit-

ted to an amplifying horn immersed in water so that the flat tip of the horn vibrates in a longitudinal direction, creating vibrational cavitation. (Because of the particular frequency involved, the term ultrasonic cavitation is also appropriate.) Test specimens are positioned directly below the horn tip in order to receive the full etching action of the cavitation bubbles. Cooling water is supplied to remove the heat generated.

In the initial work by Young, obliterated numbers on specimens fabricated from copper, brass, steel, and aluminum were investigated (4, 5). Complete or partial restorations were ultimately obtained for all metals considered. Subsequent work explored applications to authentic specimens involved in police work (6). Restorations were successfully accomplished on steel gun parts, but were only partially successful on an aluminum motorcycle gear case housing. No number recovery was achieved with a heavily obliterated and restamped cast iron automobile engine block. Advantages cited for the ultrasonic method are 1) its applicability without variation to all metals, and 2) the elimination of chemical reagents and preliminary surface treatment of the specimen.

LABORATORY PROCEDURES

Two ultrasonic generating systems—both commercially available—were used in the present work for inducing cavitation in water. These units and their specifications are as follows:

Model:	Bronwill Biosonik IV Ultrasonic System	Sonifier W-350 Cell Disruptor
Manufacturer:	VWR Scientific P..O. Box 3200 San Francisco, Calif. 94119	Branson Sonic Power Co. Eagle Rd. Danbury, Conn. 06810
Transducer:	lead zirconium titanate 20-kHz frequency	lead zirconium titanate 20-kHz frequency
Power Supply:	variable	variable

Maximum Power
During
Cavitation: 225 watts   250 watts

Horn: 3/4-inch titanium   1/2- and 3/4-inch
                       alloy probe   titanium alloy
                                                    disruptor horns

The first of these systems is shown in Figure 4-1. As shown, tap water
was continuously circulated into the water bath. This bath was supported by
a lab jack so that a specimen in the bath could be conveniently raised to any
distance below the horn tip.

Laboratory specimens were stamped and obliterated as described in
Chapter 2. The were clamped to a weighted base and submerged in the
water bath for restoration testing. The distance from horm tip to specimen
was fixed by use of removable spacers. Specimens were subjected to cavit-
ation etching for consecutive intervals of 1 minute or more and visually ex-
amined after each interval for percent of number recovered.

RESULTS AND DISCUSSION

Preliminary experiments were conducted to optimize the ultrasonic
cavitation method with respect to experimental parameters. The results
showed that serial number recovery can be accomplished with less time
as either the power supply output is increased or the distance from horn
tip to specimen is decreased. Both changes result in more vigorous
etching of the specimen. No conditions were found in which restoration
effectiveness was impaired by too vigorous treatment. For pratical
reasons a power output of 220 watts and separation distance of 1.3 mm
were selected as standard conditions for subsequent work.

Two variations of the method were considered in detail In the first
of these the specimen was polished prior to etching treatment, as is

customary in other restoration methods. In the second, the specimen
was not polished before treatment. The latter procedure follows that of
the NASA work. Results are given in Table 4-1 for the eight metals of this
study. The data show that specimen polishing prior to treatment improves
the restoration method with respect to both effectiveness and time required.
A correlation can be seen between time required and hardness of the metal
(Table 2-1), that is, harder metals require a longer time. This result
might be expected, since softer metals are also found to be etched more
rapidly than harder metals. The correlation is better in the case of
polished specimens.

Inspection of specimens during the course of restoration reveals
two distinct modes of recovery. The particular mechanism operating for
a given case depends upon the amount of metal removed during obliteration.
Figure 4-2 shows a time sequence in the restoration of a just-obliterated
number on low-carbon steel. It is clear that recovery is accomplished
because the number becomes pitted more rapidly than its background. As
described in Chapter 2, low-carbon steel is a metal for which just-oblitera-
tion occurs at a removal depth less than 1.00. Apparently the restoration
mechanism operating here is one in which cavitation removes smeared
metal imbedded in the base of the not completely removed number.

When a removal depth of 1.00 or greater is attained, a second mech-
anism must be in effect. Figure 4-3 shows a just-obliterated brass speci-
men after 20 and 150 minutes of treatment. Brass is a metal for which
just-obliteration is not reached up to a removal depth greater than 1.00.
In this example, restoration is accomplished because deformed metal below
the number is etched less rapidly than its surroundings. This is particu-
larly evident after 20 minutes; the number is less hazy than its background.

Clearly this is not removal of imbedded metal. Instead, the mechanism is based upon the fact that cold-working causes hardening in the metal below the number. After complete removal of the number, the hardening remains and is more resistant to cavitation etching than the background.

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Table 4-1

Ultrasonic Cavitation Etching Method Restoration Results

	Polished specimen[a]		Unpolished specimen[a]	
	Percent recovered of just-obliterated specimen	Time required	Percent recovered of just-obliterated specimen	Time required
Alloy steel	100%	30 min	85%	210 min
Cast iron	100%	30 min	80%	120 min
Low-carbon steel	100%	5 min	100%	20 min
Tool steel	100%	15 min	100%	80 min
Stainless steel	100%	7 min	100%	130 min
Aluminum alloy	70%	1 min	85%	14 min
Brass	100%	10 min	95%	150 min
Zinc alloy	100%	1 min	0%	150 min

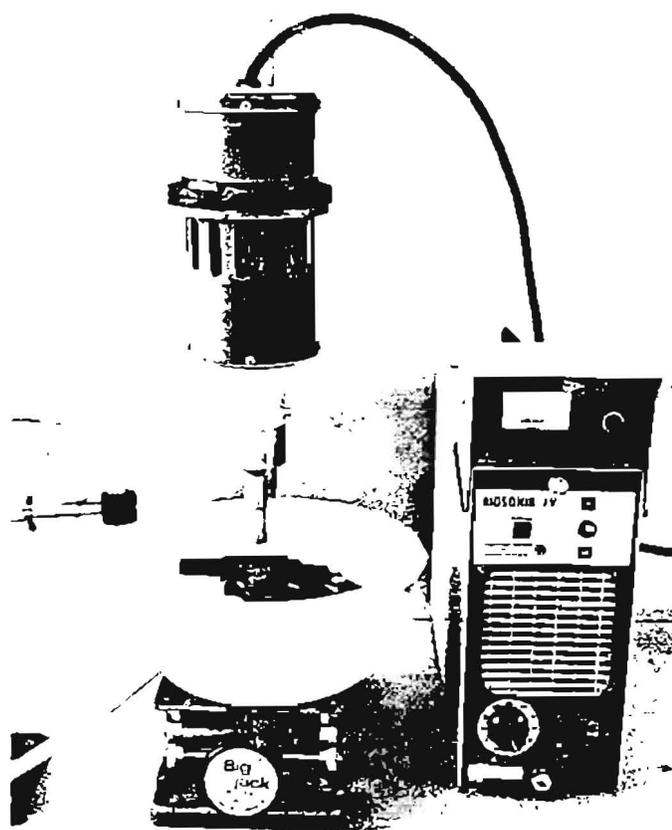[a]Ultrasonic power supply output 220 watts and horn-tip-to-specimen distance 1.3 mm.

Figure 4-1. Ultrasonic apparatus for producing cavitation in water for serial number restoration.

Obliterated
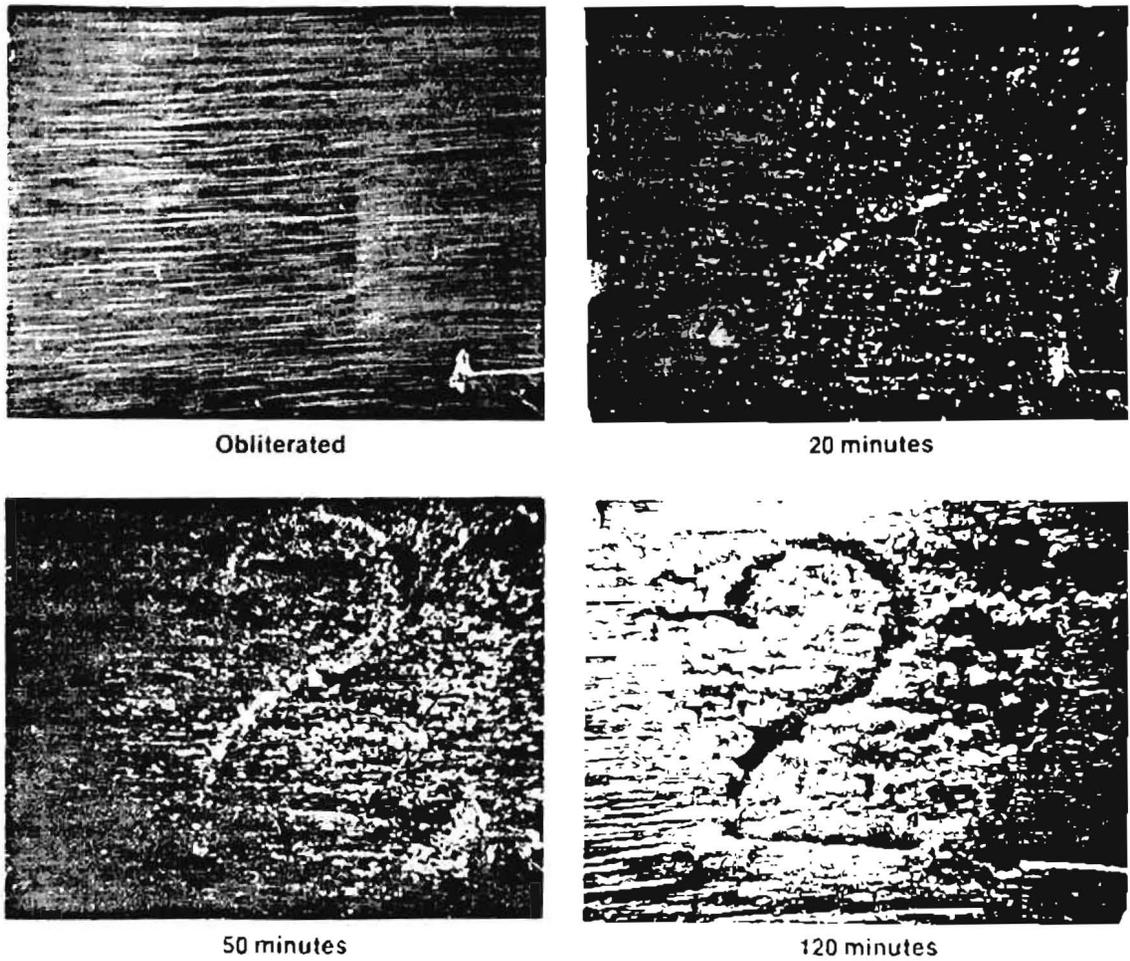
20 minutes

50 minutes

120 minutes

Figure 4-2. Restoration sequence by ultrasonic cavitation method on just-obliterated low-carbon steel (8X). Total time of treatment is indicated for each figure. Power supply output was 120 watts; horn-tip-to-specimen distance was 1.3 mm.
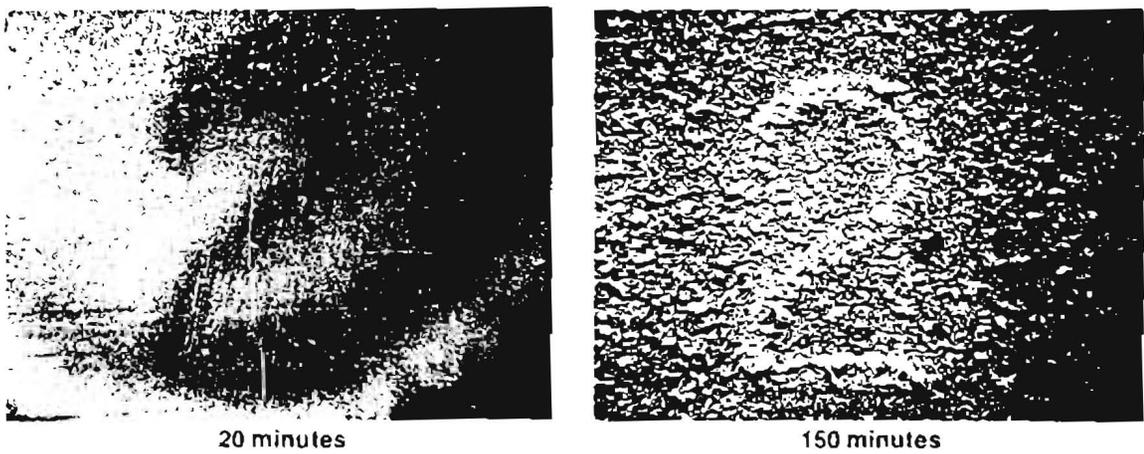
20 minutes   150 minutes

Figure 4-3.   Ultrasonic cavitation restoration on brass (8X).   Total time
of treatment is indicated for each figure.   Power supply output was 120 watts;
horn-tip-to-specimen distance was 1.3 mm.

CHAPTER 5

MAGNETIC PARTICLE METHOD

INTRODUCTION

A technique frequently used by metallurgists to detect surface or
subsurface flaws in iron or steel is known as magnetic particle inspection
(1, 2). In this technique the test specimen is first magnetized and then
sprayed with finely divided magnetic particles. The particles migrate
on the surface and accumulate at the point where a crack or other discontin-
uity exists.

A variety of magnetic particle compositions are commercially pro-
duced for inspection work. They are available as dry powders or suspen-
sions in either an oil or water vehicle. In one variation the particles are
coated with a chemical that fluoresces when inspected under ultraviolet
light. Methods used in industry for temporarily magnetizing the specimen
involve one of the following: 1) passing high-amperage direct current from
a magnetic testing unit through the test piece, 2) placing the piece in contact
with a yoke magnet, or 3) inserting it in a coil electromagnet. Figure 5-1
illustrates these magnetization methods.

Application of magnetic particle inspection to restore serial numbers
was reported in 1950 by the FBI (3, 4). As with other restoration methods,
the specimen is first polished. It is then placed between the contact plates
of a magnetic testing unit. Because the specimen is an electrical conductor,
only a low voltage need be applied to accomplish the required high-amperage
current flow. Magnetic particles applied to the specimen outline the obliter-
ated number if the restoration is successful. The magnetic particle method
still remains in use by the FBI today (5). Because it is nondestructive to
the specimen surface, it can be attempted first without hindering subsequent
restoration work by other methods.

In 1960 Wolfer and Lee suggested a simpler method for magnetizing the specimen (6). It is placed in direct contact with a large permanent horsehoe magnet with the obliterated area directly between the poles. To facilitate migration of the particles over the surface, these authors suggest touching a vibrator to the specimen.

LABORATORY PROCEDURES

Specimens were prepared and polished as described in Chapter 2. Two methods of magnetizing specimens were investigated. They were: a large horseshoe magnet (shown in Figure 5-2), and a commercial magnetic testing unit passing 1000 amperes of direct current. Three magnetic particle compositions available from Magnaflux Corporation of Chicago, Illinois were tested for effectiveness:

(1) Gray Powder: a general-purpose high contrast, dry powder.

(2) 9CM Red Spray: an oil suspension of reddish brown powder in aerosol can.

(3) 14AM Fluorescent Spray: an oil suspension of bright yellow-green fluorescent powder in aerosol can.

All compositions were applied as a fine spray. A high-intensity ultraviolet lamp was used for the inspection of the fluorescent spray.

RESULTS AND DISCUSSION

The magnetic method was applied to just-obliterated specimens of all ferrous metals of this study. Results are summarized in Table 5-1. Generally the obliterated numbers were either totally recovered or not at all. All three magnetic particle compositions were tested with the horseshoe magnet. Fluorescent spray with ultraviolet-lamp inspection gave the best results in that numbers were recovered with greatest clarity. Red spray gave quantitatively the same results, but quality was poorer. Gray powder was totally ineffective. Because of its superiority the fluorescent spray was also used in tests with the magnetic testing unit. Results obtained were no better than with the horseshoe magnet with the minor exception of cast iron. In view of its high cost and space consumption, the magnetic testing unit cannot be recommended for restoration purposes.

Figure 5-3 shows numbers restored on alloy and stainless steel. As illustrated, the specimen can be positioned with the magnetic field running vertically or horizontally. For alloy steel it is apparent that portions of the number oriented parallel to the magnetic field are not restored. All other portions are outlined by the magnetic particles. Restorations on low-carbon steel and tool steel have this same appearance. Stainless steel is exceptional in that the particles accumulate directly on the number rather than outlining it.

The theoretical basis for the magnetic particle method is illustrated in Figure 5-4. At the point of a deformation running perpendicular to the magnetic field a magnetic "leak" occurs. This leak creates localized north and south poles at opposite edges of the deformation. Magnetic particles are attracted to the poles and, thus, create the outline seen on restorations. This mechanism applies to alloy steel, low-carbon steel, and tool steel. Stainless steel is exceptional in that the austenitic type used in this study is nonmagnetic in its normal state. It is known, however, that localized regions of austenitic stainless steel can become magnetic with cold-working. Thus, restorations are accomplished not from a magnetic leak but because deformed metal under the number is weakly magnetized and, thereby, attracts particles.

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Table 5-1

Magnetic Particle Method Restoration Results

	Percent recovered of just-obliterated specimen			
	Horseshoe magnet[a]			Magnetic testing unit[a]
	Fluorescent spray	Red spray	Gray powder	Fluorescent spray
Alloy steel	100%	100%	0%	100%
Cast iron	0%	0%	0%	40%
Low-carbon steel	100%	100%	0%	0%
Tool steel	100%	100%	0%	100%
Stainless steel	100%	100%	0%	100%

[a]Method of specimen magnetization.

(A)

Magnetic field

Electric current

(B)

N S

Magnetic field

(C)
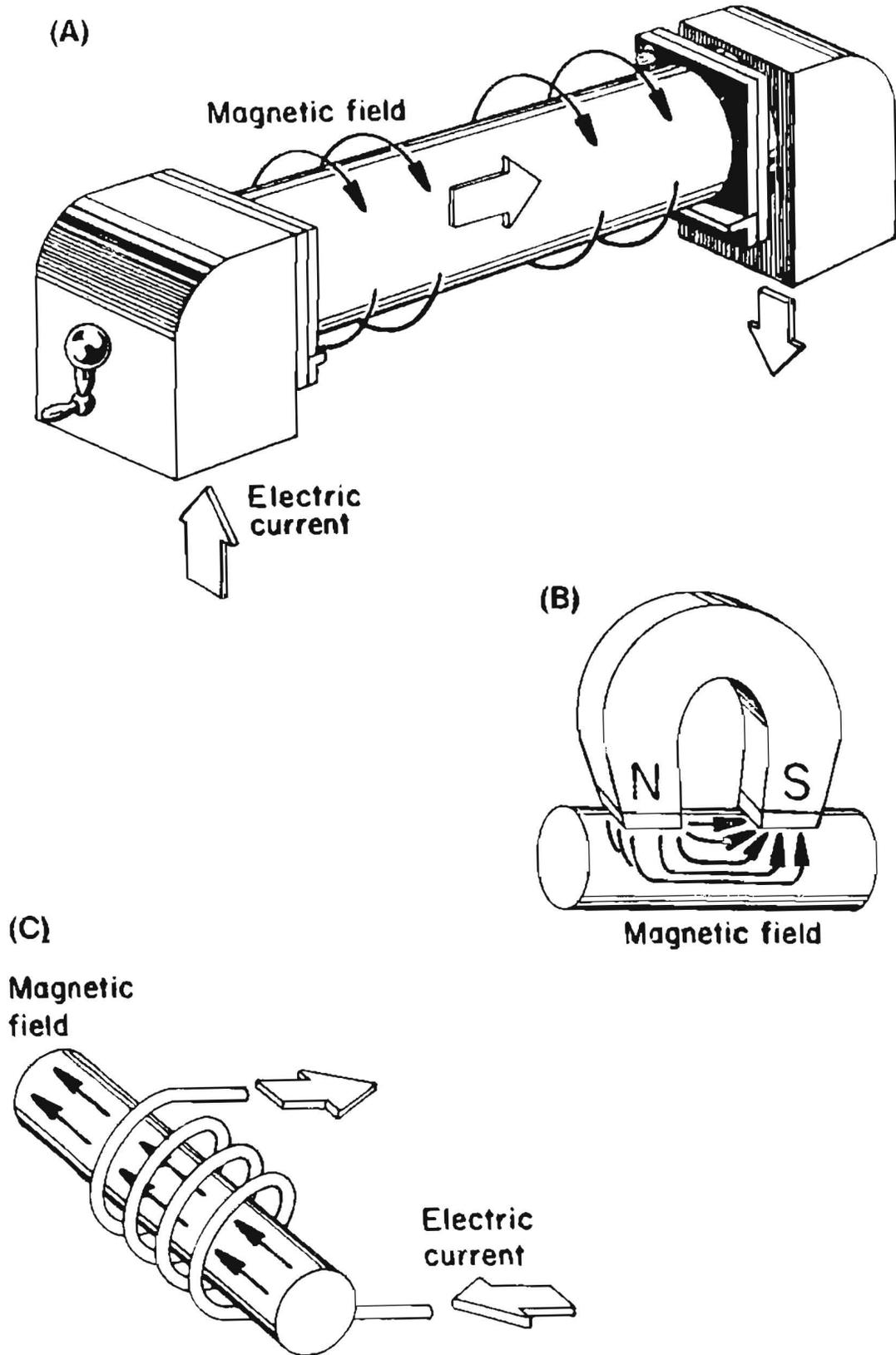
Magnetic field

Electric current

Figure 5-1. Methods of specimen magnetization. (A) Magnetic testing unit passing high-amperage direct current. (B) Yoke or horseshoe magnet. (C) Coil electromagnet.

Figure 5-2. Magnetic restoration method.
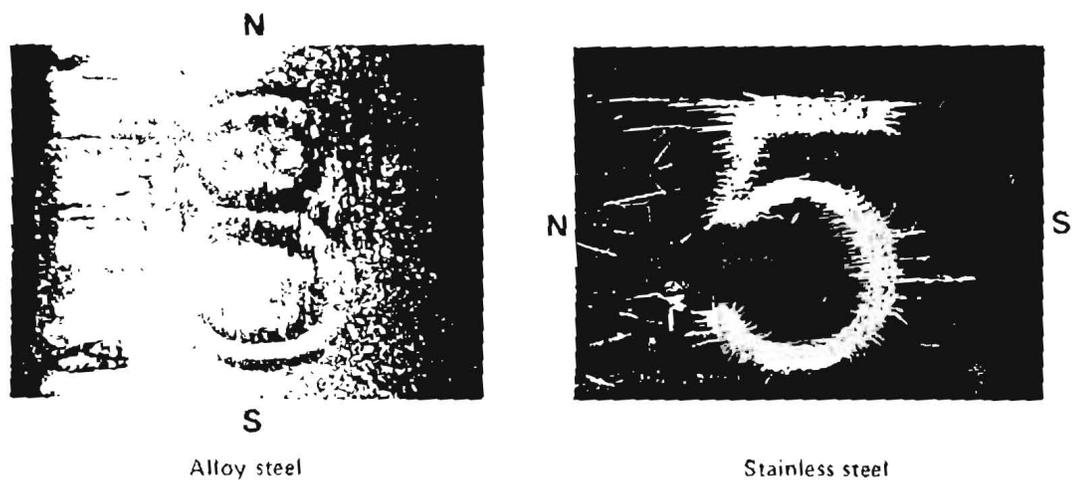
Alloy steel   Stainless steel

Figure 5-3. Magnetic particle method restorations on alloy steel and stainless steel (8X). 9CM red spray was used with magnetization by horseshoe magnet. (N and S indicate orientation of magnetic field.)
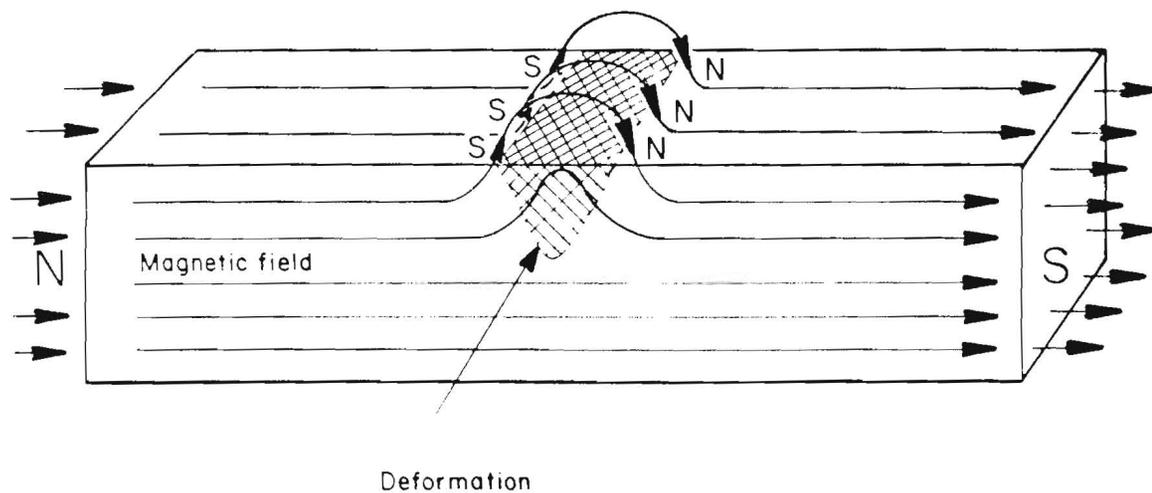
Figure 5-4. Magnetic north and south poles created at opposite edges of a deformation by leakage of magnetic field from specimen

CHAPTER 6

HEAT TREATMENT AND OTHER METHODS

HEAT METHOD INTRODUCTION

The various phases present in a polycrystalline metal will display
different behavior when subjected to heat. Use can be made of this fact to
investigate the microstructure of the metal. In the technique of heat tinting,
a specimen is heated in the presence of air until a colored oxide film forms
over its surface. This coating may reveal structural features of interest.
The process of heat etching involves heating the specimen to still higher
temperatures, but in an inert atmosphere or vacuum. Eventually, vaporiza-
tion occurs within certain phases or phase boundaries, enabling the metal
microstructure to be observed. Neither of these techniques is extensively
used in metallography, but both are briefly mentioned in the reference work
of Kehl (1).

The use of heat treatment to restore serial numbers was described
by the FBI in 1956. According to the procedure given, a welding torch is
used to heat the specimen until the obliterated number rises from the sur-
face and becomes visible (2). The National Auto Theft Bureau recommends
this technique on cast iron engine blocks (3). According to this latter
reference the metal is first given a mirror-like polish and is then heated
very gradually to accomplish restoration. After cooling, the surface is
lightly polished with oiled emery paper to remove deposited carbon. The
number is said to appear lighter in color than its surroundings at this point.

LABORATORY PROCEDURES

Specimens were heated with an oxygen natural gas torch (see Figure
6-1) using two distinct techniques. In the procedure referred to as indirect
heat, the obliterated area was warmed gradually by conduction, with the

The nonferrous metals behaved as follows:

Aluminum alloy:	100% restoration; the surface dulled and small cracks formed. The number appeared as more heavily cracked.
Brass:	100% restoration; the color changed to gold, violet, silver, and yellow. The number was visible as dark yellow.
Zinc alloy:	100% restoration; the surface dulled and number appeared as sunken below surface. Visibility was lost when melting occured.

Direct Heat. — The specimens were heated until melting began or until the surface glowed red. Results obtained were:

Alloy steel:	No restoration: a black oxide formed upon cooling; polishing was ineffective at revealing the number.
Cast iron:	100% restoration; the number appeared as raised while red-hot; a black oxide formed upon cooling.
Low-carbon steel:	100% restoration; the color changed to violet and blue and finally, red-hot; a gray oxide formed upon cooling. Polishing was ineffective at revealing the number.
Tool steel:	100% restoration; the color changed to gold, violet, silver and, finally, to a dull surface. The number appeared as less dulled.
Stainless steel:	100% restoration; the color changed to gold, violet, dark violet, and silver. Finally, it dulled except for number, which retained shine.
Aluminum alloy:	Partial restoration: the surface dulled, blistered, and melted. The number was visible as ridges of blisters.

Brass:	No restoration; the color changed to gold, violet, silver, yellow, blue-green, and finally, orange-hot; a gray-black oxide formed upon cooling. Polishing was ineffective at revealing the number.
Zinc alloy:	100% restoration; the surface dulled and number appeared as sunken below surface; blistering and melting resulted in loss of restoration.

The results show that indirect heating is more effective than direct, since the former was successful on all metals.

OTHER METHODS

The criminal science literature contains brief mention of restoration methods in addition to those given detailed consideration in the present work. These methods are less well documented and in some cases may simply represent speculation on the part of the author. A few have been briefly investigated in this work.

Cold-Frost Method

It has been reported that restoration can be accomplished by selective deposition of frost on an obliterated number (4, 5). According to this method the specimen is first polished and then wiped over with Dry Ice. It is cooled to the point where atmospheric water vapor frosts the surface. If recovery is successful, the number is made visible by the frost pattern.

In this laboratory, attempts to restore numbers by the cold-frost method were unsuccessful. The specimen used was just-obliterated alloy steel for which restoration by other methods could have been easily accomplished. Frost deposition on the surface did not recover the number. To further explore the effectiveness of the general method, a second alloy steel specimen was subjected to iodine (I_2) vapor until a coating of that element sublimed onto the obliterated area. Once again no restoration was obtained.

Radiography

X-rays and gamma rays will penetrate a metal to an extent determined by the density and the atomic number of the atoms encountered. Using a photographic plate sensitive to transmitted radiation, a radiograph of a

metal specimen can be made just as an X-ray photograph can be made of the human body. Metallurgists commonly use this technique to inspect castings, weldings, and forgings for defects. Application of radiography to serial number restoration appears never to have been reported, but the concept has been suggested (4, 5).

To test the method, radiographs were made of just-obliterated specimens of all eight metals of this study. No evidence of the obliterated numbers was revealed. Apparently radiography lacks adequate sensitivity to detect the deformation below an obliterated number.

Radiographs taken of the specimens prior to obliterating did clearly show the numbers. This indicates the technique is potentially useful for locating concealed serial numbers. These would include a hidden number on a firearm or one concealed by layers of paint.

Liquid Penetrant Method

Liquid penetrant testing is a common industrial technique for locating invisible cracks in metals. The method is non-destructive and, unlike the magnetic particle method, is applicable to both ferrous and nonferrous metals (6). The literature contains no reference to the use of liquid penetrants for recovering serial numbers.

To determine its effectiveness, the liquid penetrant method was tested on stainless steel, aluminum alloy, brass, and zinc. Specimens were just-obliterated and polished in the conventional fashion. A commercially available fluorescent penetrant, Zyglo®ZL-22A, and a developer, ZP-9, were employed. (These products are manufactured in aerosol form by Magnaflux Corporation of Chicago, Illinois.) After the specimen surface was cleaned, it was sprayed with penetrant. Penetration times of 5 to 30 minutes were investigated, after which time the excess penetrant was gently rinsed away with a fine water spray. A fine spray of developer was next applied. The role of the developer is to draw to the surface any penetrant present in cracks and to act as a contrasting background for the observation of this fluorescing material. After drying, the specimen was inspected under a

high-intensity ultraviolet lamp. For none of the four metals investigated
did this procedure accomplish recovery of the obliterated number. No
further testing was done.

Electroplating

Electroplating is the reverse process of electroetching. An external
dc power source, such as shown in Figure 3.2. is used to induce build-up
of a metallic coating on a specimen surface. The specimen is connected by
a wire to the negative (-) terminal of the power source, and a cotton swab
wetted with electroplating solution is connected to the positive (+) terminal.
When the swab is brushed across the specimen. metal ions from the solu-
tion are plated onto the surface. If electroplating of deformed metal below
an obliterated number is sufficiently different from that of its surroundings,
the number will be recovered. The possibility of accomplishing restorations
in this way has been suggested (4, 5).

In order to test the method. numbers were stamped on alloy steel
and removed to just-obliteration. The obliterated areas were polished and
platings were applied using commercial electroplating solutions. The
platings tested were copper, brass and nickel. In no case was restoration
accomplished. Electroplating occurred too uniformly to enable the obliter-
ated numbers to be distinguished.

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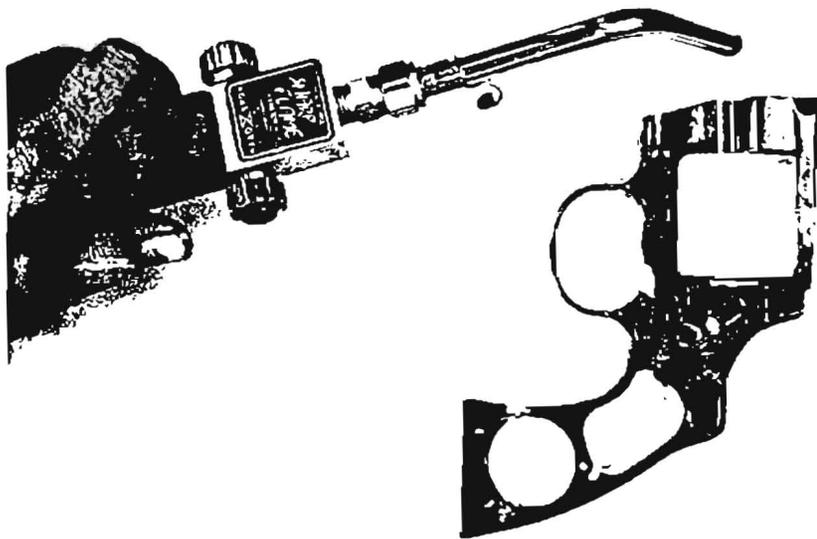
Figure 6-1. Heat treatment restoration method

CHAPTER 7

EVALUATION OF METHODS

In preceding chapters restoration methods were evaluated according to their ability to recover numbers obliterated just to the point of not being visible. Each of the major methods developed proved effective in at least one variation at recovering just-obliterated numbers. Thus, the test results do not enable meaningful conclusions as to their relative values. In the crime laboratory the investigator commonly encounters specimens for which removal of metal is beyond the minimum necessary for just-obliteration. For the purpose of differentiating the restoration effectiveness of the various methods, tests were conducted to determine their abilities to recover numbers from depths beyond just-obliteration.

To evaluate any selected method, data were obtained on the percent of number recovered as grinding depth was increased. Figure 7-1 shows results obtained for alloy steel. It consists of graphs of percent recovered versus removal depth for five restoration methods. As defined in Chapter 2, removal depth is the ratio of the thickness of metal removed by grinding to the stamping depth of the number. When the removal depth is near 1.00, all methods illustrated in Figure 7-1 are successful at recovering the number. Deeper grinding results in more complete removal of deformed metal and, thus, places greater demands on the restoration method. Eventually, each of the methods fails, and its percent-recovered plot drops to the zero baseline. Figure 7-1 shows that the electrolytic method using Turner's reagent is the most effective on alloy steel.

For the purpose of summarizing large amounts of data a numerical parameter derived from graphs of percent recovered versus removal depth proves useful. The parameter is defined as the removal depth at which the portion recovered has a value of 50%. It is the half-recovery removal

depth and is symbolized RD_{50}. In Figure 7-1 the RD_{50} values for methods A thru E are 1.15, 1.40, 2.15, 3.00, and 3.10, respectively. In cases where duplicate experiments were performed, RD_{50} values were reproducible to within ±10%.

IRON AND STEEL

Only those restoration procedures judged to be most promising in earlier testing were evaluated at greater obliteration depths. In general, those procedures based upon etching of the metal were found to require more etching time as obliteration depth increases. Thus, a chemical-method restoration that could be accomplished in less than one minute at just-obliteration might take ten minutes at a removal depth of 2.00.

Table 7-1 lists RD_{50} values obtained on iron and steel. Values range from less than 1.00 to a maximum of 5.90. (Values of less than 1.00 can result for a specimen in which just-obliteration occurs at a removal depth less than 1.00 and no recovery is obtained beyond that depth.)

The chemical and electrolytic methods generally proved more effective than other methods on the ferrous metals. These metals vary considerably in their susceptibility to chemical attack (see Chapter 3). Thus, it is not surprising that a mild chemical etchant such as ferric chloride, which is effective on low-carbon steel, is ineffective on the other ferrous metals. A more vigorous etchant is 25% nitric acid. It reacts too rapidly to be effective on low-carbon steel, but does well on alloy steel, cast iron, and tool steel. It is too inert on stainless steel. One group of chemical etchants was found to be universally effective on the ferrous metals. The group consists of the three etchants containing cupric compounds and hydrochloric acid, as typified by Fry's reagent. These etchants characteristically give restorations of good clarity and high RD_{50} values on all ferrous metals. Turner's electrolytic reagent is also a member of this chemical group. On some metals its use as an electrolytic etchant gave slightly greater RD_{50} values than Fry's reagent as a chemical etchant. It also requires less reacting time.

As seen in Table 7-1, methods other than chemical and electrolytic etching generally give inferior results. Notable exceptions are the superiority of the indirect heat method on cast iron and the magnetic method on tool steel. The data also show that in the magnetic particle method the fluorescent spray is slightly preferable to the nonfluorescent. With the ultrasonic cavitation method, a benefit from polishing the specimen prior to treatment is seen in the two metals so considered.

For all of the ferrous metals, numbers could be recovered from removal depths of at least three times the stamping depth. This is an indication of the depth to which crystal grain deformation extends. The deepest recovery was obtained using Turner's reagent on stainless steel. Stainless steels are known to be more susceptible to cold-working than other steels. Metallographic examination shows that subtle evidence of deformation exists at great depths below a stamped impression. As illustrated in Figure 7-2, extensive grain compression occurs at the base of the impression (A) and also 1 mm below it (B). At 2 mm below the impression the grains are not compressed, but an occasional grain deformation by slip can be seen (C). (A distance of 2 mm below the impression corresponds to a removal depth of about 7.7)

NONFERROUS METALS

In a similar fashion, restoration methods previously judged most promising on aluminum alloy, brass and zinc alloy were tested at depth. Table 7-2 lists RD_{50} values obtained for these nonferrous metals.

On aluminum alloy the most effective method found is electrolytic etching with ferric chloride, which gave an RD_{50} of 2.40. The two best etchants by the chemical method were acidic ferric chloride and hydrofluoric acid in mixed acids. The ultrasonic cavitation and indirect heat methods were slightly less effective. However, no method was found to give recoveries of the good clarity typical of those on ferrous metals.

Several etchants of the chemical method gave good results on brass. The three most effective were acidic ferric chloride, chromic acid, and 25% nitric acid. All gave recoveries judged to be of good clarity, with RD_{50} values of about 2.60. Acidic ferric chloride produces results in the least time. Use of acidic ferric chloride as an electrolytic etchant did not improve its effectiveness, and 25% nitric acid lost effectiveness because of too rapid etching when given the electrolytic assist. The ultrasonic cavitation method is competitive with the chemical and electrolytic methods on brass.

The most effective restoration method found for zinc alloy is chromic acid as a chemical etchant. It gave an RD_{50} of 3.50. Slightly less effective is 10% sodium hydroxide by the electrolytic method. The ultrasonic cavitation and indirect heat methods ranked below these. As with aluminum no method gave recoveries judged to be of good clarity.

The nonferrous metals, particularly aluminum alloy and brass, did not yield RD_{50} values as high as those of the ferrous metals. Crystal grain deformation apparently does not occur to the same depth for the nonferrous metals. A variety of factors probably create this situation. The nonferrous metals are less hard and, therefore, accept a stamped indentation with transmission of less stress through the specimen. In addition, the aluminum alloy and brass laboratory specimens were wrought metal. This means that they contained considerable cold-working as initially received and that deformation due to stamping had to be superimposed upon an already present background. Different results would be expected on annealed specimens.

HANDGUNS AND OTHER AUTHENTIC SPECIMENS

All results reported heretofore were obtained on specimens prepared in the laboratory specifically for restoration testing. Such specimens are well suited for the determination of restoration effectiveness because of the precision which can be achieved in stamping and removal depth. In the present section results obtained earlier are tested on authentic specimens of the type encountered in the crime laboratory.

Restoration testing was done on serial numbers obliterated from two types of Colt handgun frames manufactured from different varieties of steel:

    (1) AISI 4140 forged alloy steel Colt Model-I revolver frames. Such frames are used in common Colt .357 Mag. revolvers such as the Law Man and Police Python models.

    (2) AISI 1137 forged medium-carbon steel Colt Model-S pistol frames. These frames are used in Colt 22 L.R. target pistols.

AISI 4140 is the same metal investigated previously in the form of alloy steel laboratory specimens. AISI 1137 medium-carbon steel is similar in properties to low-carbon steel except for its lower chemical reactivity and greater hardness.

Serial numbers on the steel handgun frames were removed to just-obliteration with a silicon grinding point. Table 7-3 gives results of restoration attempts using selected methods. For the alloy steel revolver frames, restorations of the greatest clarity were obtained with Fry's reagent, acidic cupric sulfate, 25% nitric acid, and Turner's reagent. The medium-carbon steel pistol frames gave greatest clarity with Fry's reagent, acidic ferric chloride, and Turner's electrolytic reagent.

In a similar fashion restoration methods previously found effective on alluminum alloy laboratory specimens were tested on authentic specimens. The authentic specimens represent two varieties of aluminum alloy with distinct properties and major alloying elements:

    (1) AA 2014 forged aluminum/copper alloy in the form of Colt Model-D light-revolver frames. These frames are used in .38 Spc. revolvers such as the Diamondback and Detective models. The alloy contains 4.4% copper.

    (2) AA 333 cast aluminum/silicon alloy in the form of a compressor housing manufactured by Bohn Aluminum and Brass Corp. The principal alloying elements are 8.0-10.0% silicon and 3.0-4.0% copper.

By contrast, the laboratory specimens used for earlier testing were of AA 6063 extruded aluminum alloy, which contains comparatively minor amounts of alloying elements (see Table 2-2).

Restoration results obtained on the revolver frames and casting are shown in Table 7-4. Because of the higher percentage of alloying elements in these specimens, their response to chemical and electrolytic etchants differs from that found earlier for laboratory specimens. In particular, treatment with hydrofluoric acid in mixed acids causes them to blacken rather than to reveal brilliant crystal grain structure, and treatment with acidic mercuric chloride does not develop the aluminum oxide coating described in Chapter 3.

The authentic specimens were less effectively restored than the laboratory specimens. This is particularly true with the revolver frames for which restoration was accomplished only by the hydrofluoric acid in mixed acids and the indirect heat method. It was found that restorations of superior clarity were obtained on the revolver frames when these two methods were used together; i. e., the specimen was first heated with a torch until the number appeared, and the surface was treated with hydrofluoric acid in mixed acids after cooling. Treatment with the chemical etchant improves the quality of the recovery obtained by heat treatment alone.

CONCLUSIONS AND FURTHER CONSIDERATIONS

From the restoration effectiveness results (Tables 7-1 through 7-4), conclusions can be readily drawn as to preferred methods for use on each metal. Such conclusions must be qualified by recognition that they represent current knowledge of the subject. Quite probably the limits of restoration effectiveness have not yet been reached. Recommendations given in the next chapter are intended to serve as points of departure for the laboratory investigator, who must be alert to unusual circumstances. In the course of serial number restoration work, a nearly infinite variety of metal alloys can be encountered. The need for thorough knowledge of the restoration process is always present.

Each of the major restoration methods is reviewed in Table 7-5. Listed with each method are those practical considerations which should be remembered, in addition to restoration effectiveness, when evaluating the method.

Table 7-1

Restoration Effectiveness at Depth on Iron and Steel[a]

| Method | RD_{50} | | | | |
	Alloy steel	Cast iron	Low-carbon steel	Tool steel	Stainless steel
Chemical[b]					
Fry's reagent	3.00	2.65	3.05	3.55	4.60
Acidic cupric sulfate	2.05	- - -	- - -	3.75	3.15
Acidic cupric chloride	- - -	- - -	3.30	1.70	- - -
Acidic ferric chloride	- - -	1.10	3.10	- - -	- - -
Ferric chloride	2.00	- - -	3.05	2.50	- - -
Ammonium persulfate	1.65	1.40	2.05	2.90	- - -
25% nitric acid	2.15	2.30	2.15	4.00	- - -
Mixed acids	- - -	1.65	- - -	- - -	- - -
10% nitric acid in ethyl alcohol	- - -	- - -	2.50	- - -	- - -
10% hydrochloric acid in methyl alcohol	- - -	- - -	2.70	- - -	- - -
Electrolytic[c]					
Davis' reagent	- - -	- - -	2.75	3.20	5.25
Turner's reagent	3.10	3.40	3.00	4.00	5.90
Arai's reagent	2.00	3.30	- - -	- - -	- - -
Ammonium persulfate	- - -	3.10	- - -	1.80	- - -
10% hydrochloric acid in methyl alcohol	- - -	3.35	2.80	2.75	- - -
Ultrasonic cavitation					
Polished specimen	1.40	3.35	2.40	3.00	2.55
Unpolished specimen	- - -	- - -	1.80	- - -	< 1.00

Table 7-1 (Cont.)

Method	RD_{50}				
	Alloy steel	Cast iron	Low-carbon steel	Tool steel	Stainless steel
Magnetic particle					
Fluorescent spray	1.15	<1.00	1.95	4.25	2.20
Red spray	<1.00	<1.00	1.40	4.00	1.90
Heat treatment					
Indirect	<1.00	5.05	1.50	3.40	3.90

[a] See text for explanation of RD_{50}.

[b] See Table 3-4 for chemical etchant formulations.

[c] See Table 3-6 for electrolytic etchant formulations.

Table 7-2

Restoration Effectiveness at Depth on Nonferrous Metals[a]

	Method	RD_{50}
Aluminum alloy	Chemical[b]	
	Cupric chloride in nitric acid	1.70
	Acidic ferric chloride	1.95
	Ferric chloride	1.50
	Acidic mercuric chloride	1.50
	Hydrofluoric acid in mixed acids	1.95
	Electrolytic[c]	
	Ferric chloride	2.40
	Hydrofluoric acid in glycerol	1.40
	Hydrofluoric acid in mixed acids	1.40
	10% sodium hydroxide	1.65
	Ultrasonic cavitation	
	Polished specimen	1.40
	Unpolished specimen	0.85
	Heat treatment	
	Indirect	1.40
Brass	Chemical[b]	
	Acidic ferric chloride	2.65
	Chromic acid	2.60
	Hydrogen peroxide	1.45
	25% nitric acid	2.60
	Electrolytic[c]	
	Acidic ferric chloride	2.55
	25% nitric acid	1.50
	Ultrasonic cavitation	
	Polished specimen	2.50
	Unpolished specimen	2.30
	Heat treatment	
	Indirect	1.05

Table 7-2 (Cont.)

	Method	RD_{50}
Zinc alloy	Chemical[b]	
	Ferric chloride	1.30
	Chromic acid	3.50
	Iodine	2.55
	5% hydrochloric acid in ethyl alcohol	1.85
	Electrolytic[c]	
	Chromic acid	3.15
	10% sodium hydroxide	3.20
	Ultrasonic cavitation	
	Polished specimen	2.75
	Unpolished specimen	1.85
	Heat treatment	
	Indirect	2.25

[a]See text for explanation of RD_{50}.

[b]See Table 3-5 for chemical etchant formulations.

[c]See Table 3-7 for electrolytic etchant formulations.

Table 7-3

Restoration Results on Colt Steel Handguns

Method	Model-I revolver frames AISI 4140 Alloy steel		Model-S pistol frames AISI 1137 Medium-carbon steel	
	Percent recovered for just-obliterated specimen	Time required	Percent recovered for just-obliterated specimen	Time required
Chemical[a]				
Fry's reagent	95%	30 sec	100%	2 min
Acidic cupric sulfate	100%	1 min	- - -	- - -
Acidic ferric chloride	- - -	- - -	100%	6 min
Ammonium persulfate	70%	8 min	- - -	- - -
25% nitric acid	100%	30 sec	100%	1.5 min
Electrolytic[b]				
Turner's reagent	100%	3 min	100%	5 sec
Arai's reagent	100%	1 min	100%	5 min
Ultrasonic cavitation				
Polished specimen	60%	10 min	100%	60 min
Magnetic particle				
Fluorescent spray	45%	- - -	0%	- - -
Red spray	50%	- - -	0%	- - -

[a]See Table 3-4 for etchant formulations.

[b]See Table 3-6 for etchant formulations.

Table 7-4

Restoration Results on Colt Aluminum Revolver
and Aluminum Casting

Method	Model-D revolver frame AA 2014 Aluminum/copper alloy		Compressor housing AA 333 Aluminum/silicon alloy	
	Percent recovered for just-obliterated specimen	Time required	Percent recovered for just-obliterated specimen	Time required
Chemical[a]				
Cupric chloride in nitric acid	0%	20 min	0%	15 min
Acidic ferric chloride	0%	15 min	100%	3 min
Ferric chloride	0%	20 min	80%	12 min
Acidic mercuric chloride	0%	20 min	0%	50 min
Hydrofluoric acid in mixed acids	80%	3 min	100%	15 sec
Electrolytic[b]				
Ferric chloride	0%	12 min	100%	18 min
10% sodium hydroxide	0%	10 min	100%	1 min
Ultrasonic cavitation				
Polished specimen	0%	240 min	100%	6 min
Heat treatment				
Indirect	100%	3 min	100%	5 min

[a]See Table 3-5 for chemical etchant formulations.

[b]See Table 3-7 for electrolytic etchant formulations.

Table 7-5

Restoration Method Characteristics

| Method	Description	Advantages	Disadvantages
Chemical	Etching by chemical solution	Simplicity, low cost, and portability	Requires chemical reagents specific for each metal and often hazardous
Electrolytic	Etching by electrolytic process	Relative simplicity and speed	Requires chemical reagents (as above) and electric power supply
Ultrasonic cavitation	Etching by action of water in state of cavitation	One procedure applicable to all metals	High initial cost and limited portability of equipment
Magnetic particle	Application of magnetic particles to specimen while magnetized	Nondestructive to specimen; may be tried prior to other methods	Applicable to magnetic metals only
Heat treatment	Gradual heating of surface with torch flame	One procedure applicable to all metals	Destroys metal deformation evidence, making subsequent restoration attempts impossible
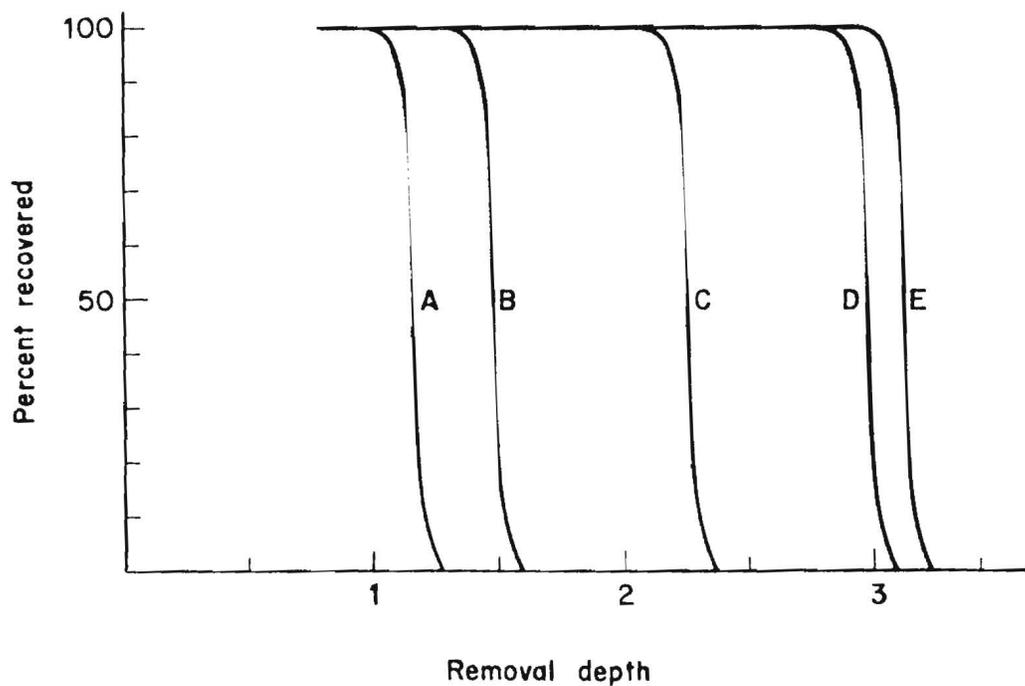
Figure 7-1. Percent of obliterated number recovered versus removal depth for selected restoration methods on alloy steel. Methods shown are: (A) Magnetic particle fluorescent spray (B) Ultrasonic cavitation (C) Chemical etching with 25% nitric acid (D) Chemical etching with Fry's reagent (E) Electrolytic etching with Turner's reagent.
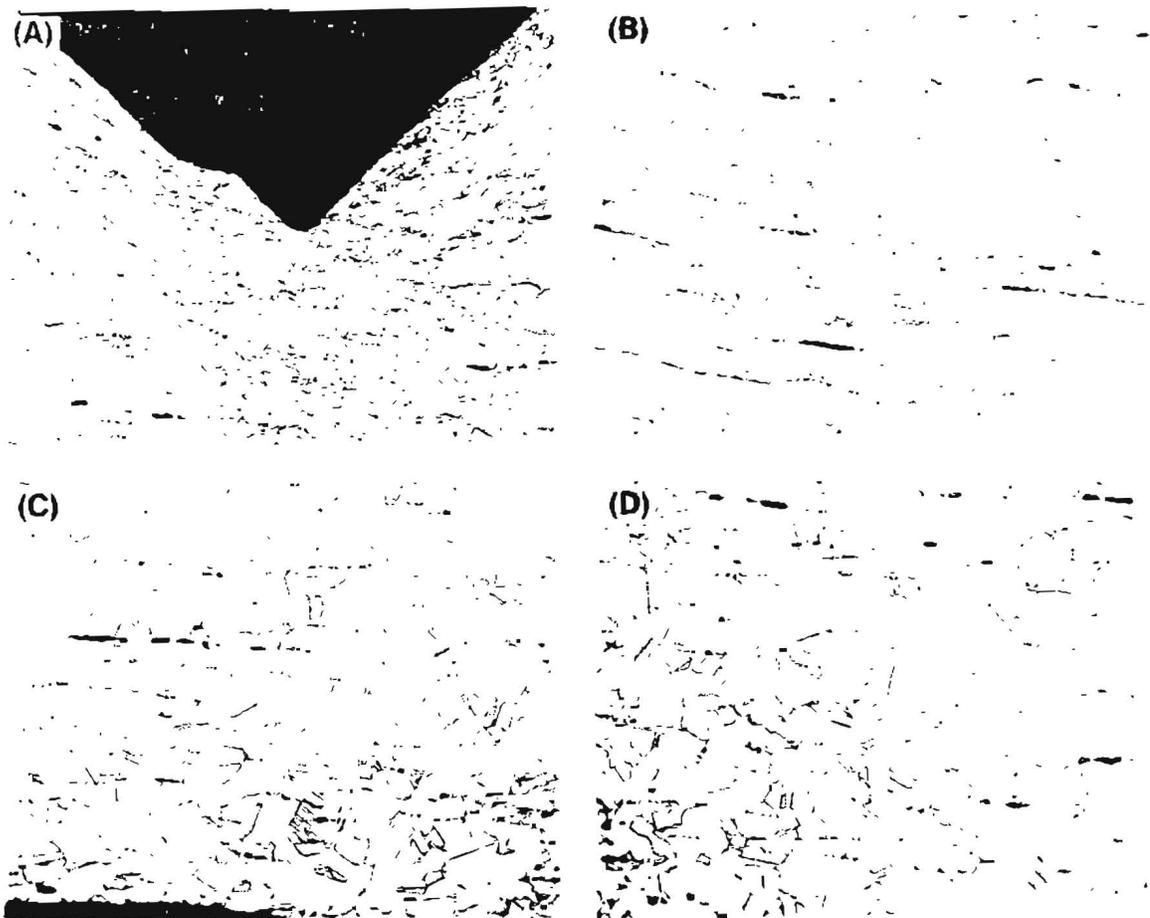
Figure 7-2. Metallographs (200X) of stainless steel specimen at various distances below number stamped to 0.3-mm depth . Exposures taken as follows: (A) At base of number (B) 1 mm below base of number (C) 2 mm below base of number (D) Totally removed from number.

CHAPTER 8

RECOMMENDED PROCEDURES

Previous   chapters   presented research findings directed toward improving and evaluating restoration methods. In order to facilitate the utilization of these findings, the present chapter outlines specific procedures recommended for use by the laboratory investigator.

CHEMICAL METHOD PROCEDURE

A major conclusion of this work is that the chemical method has much to recommend it in terms of simplicity and effectiveness. A step-by-step description of its general procedure is given here:

> Step 1. Cleaning and Polishing. First, remove grease, paint, and dirt from the specimen by application of suitable solvents, such as penetrating oil or mineral spirits. Second, polish the surface to a mirror-like finish with emery cloth, polishing rouge, or other fine abrasive. A hand-held power tool as shown in Figure 3-1 is useful for polishing. (Care must be taken not to overheat the specimen in the case of low-melting aluminum and zinc alloys.) An occasional deep scratch or pit may be allowed to remain after polishing. Such is advisable in a case where polishing to complete removal would destroy an excessive amount of the deformed metal upon which restoration depends.

> Step 2. Application of Chemical Etchant. Apply a film of etchant to the polished surface with a cotton swab, dropper, or glass stirring rod. Time required for restoration may be a few seconds to an hour, depending upon the chemical reactivity of the specimen. Occasionally during the etching process the etchant should be wiped off and reapplied. Improved clarity is often achieved by rinsing with a spray of acetone, allowing the surface to dry, and lightly rubbing or polishing.

Step 3. Observation of Restoration. The specimen must be carefully observed throughout the etching process, since restoration can occur at any time and may be lost with continued etching. Best viewing of a recovered number is accomplished under bright light with the angle of illumination varied. Magnification may be helpful for small specimens.

Figure 8-1 illustrates application of the chemical method to a revolver frame.

TECHNIQUES FOR SPECIFIC METALS

The following sections list specific reagents recommended for the chemical method and present alternative methods competitive with chemical etching for each metal type. Each of these more specialized methods requires the procedures outlined in Step 1 and Step 3 above. Further detail on their application can be found in earlier chapters.

Steel. — The many varieties of steel, from low-carbon to stainless, differ considerably in their chemical reactivity. Some etchants, such as 25% nitric acid, are effective on only certain steels. Fry's reagent is effective on all varieties:

$$90 \text{ g} \quad CuCl_2$$
$$120 \text{ ml} \quad HCl$$
$$100 \text{ ml} \quad \text{water}$$

The reagent is stable in storage and requires no special handling precautions beyond those of an acid solution. The only method typically found more effective than Fry's reagent is the electrolytic method with Turner's reagent:

$$2.5 \text{ g} \quad CuCl_2$$
$$40 \text{ ml} \quad HCl$$
$$24 \text{ ml} \quad \text{ethyl alcohol}$$
$$30 \text{ ml} \quad \text{water}$$

In the electrolytic method the specimen is cleaned and polished as above, and the etchant is applied as described in Chapter 3 and illustrated in Figure 3-2. A common 6-volt battery is a convenient dc power supply.

Cast Iron.— Fry's reagent and Turner's electrolytic reagent also work well on cast iron. However, the indirect heat method is superior to either of these etchants. (See Chapter 6) The specimen is cleaned and polished and then gradually heated with a torch applied a short distance from the obliteration. The number appears as a raised image just before the surface

becomes red hot.

Aluminum Alloys. — No single method has been found most effective on all aluminum alloys. If the investigator has no prior experience with the specimen in question, the first restoration attempt should be with the chemical etchant acidic ferric chloride:

    25 g $FeCl_3$
    25 ml   HCl
    100 ml   water

or the electrolytic etchant ferric chloride:

    25 g $FeCl_3$
    100 ml   water

Both have been shown effective on extruded aluminum alloy (AA 6063) and cast aluminum/silicon alloy (AA 333).

If the above treatments are not successful, the method found most effective on forged aluminum/copper alloy (AA 2014) should be attempted. This is the indirect heat method. (See Chapter 6.) For this technique care should be taken not to overheat the specimen to the point where it softens or becomes deformed. For photographic purposes the clarity of the indirect-heat method restoration can be improved by subsequent application of hydrofluoric acid in mixed acid etchant:

    2 ml   HF
    24 ml   HCl
    12 ml HNO_3
    2 ml   water

This reagent requires special handling, since hydrofluoric acid solutions etch glass and can create hazardous pressures in sealed containers. A vented, acid-resistant plastic bottle is recommended with storage limited to a few days.

Brass. — For effectiveness and simplicity two chemical etchants are recommended on brass. They are acidic ferric chloride:

    25 g $FeCl_3$
    25 ml   HCl
    100 ml   water

and 25% nitric acid:

    25 ml HNO_3
    75 ml   water

Both are stable in storage; acidic ferric chloride requires less time for etching. The ultrasonic cavitation method with polishing is equally effective on brass. (See Chapter 4.)

PRACTICAL SUGGESTIONS

Identification of Metal Alloys

Selecting the best bethod for accomplishing a restoration requires knowledge of the particular metal from which the specimen is made. A great number of metal alloys are produced commercially today. The complete analysis of a metal to establish its detailed identity requires a major laboratory effort. Fortunately, in its current state of refinement serial number restoration work requires only identification of the broad classification to which the metal belongs.

Numerous laboratory techniques are available for metal analysis. Perhaps the simplest of these involve placing a drop of chemical reagent on the specimen and observing reactions which occur, particularly the development of colored spots. Such tests are known as spot tests. Spot tests have been developed for a variety of applications (1, 2). A scheme is suggested here which is very easy to perform and should prove adequate for present purposes. It consists of 1) testing the metal to determine if it is magnetic and 2) applying one drop of concentrated nitric acid to the surface cleaned with emery cloth for ten minutes and then rinsing.

This scheme has been applied to the specimen metals of the present study and several others of interest. Results are given in Table 8-1. Because reactions will vary with conditions, it is recommended that the investigator first apply the spot test to known metals to gain experience before using it for unknowns. Certain additional characteristics of the metals are useful for identification. Thus, aluminum is unique in its low density and its initiation of effervesence upon treatment with concentrated solutions of sodium hydroxide. Lead has a higher density and is softer than all other common metals.

Styles of Numerals Used for Serial Numbering

Even with the best techniques, a restoration attempt may recover only a portion of an obliterated numeral. To get the most information from a partial recovery, the investigator should have knowledge of the styles of numerals used for serial numbering. Figure 8-2 shows styles of numerals used by twelve handgun manufacturers. Considerable variety of style can be seen. For example, there are four distinct types of numeral 1:

$$ 1 \quad 1 \quad 1 \quad | $$

The first and third of these could easily be mistaken for an incompletely restored 4. Similarly, the numerals 3 and 8 can be confused. Note that most manufacturers use 6-digit serial numbers, but 5-digit and 7-digit numbers can also be found. The reference work of Krcma contains many illustrations showing serial numbering styles (3). Cbviously, a manufacturer may change styles at any time.

Recording Results

The chances of achieving a successful serial number restoration are increased if the investigator takes care to record all observations made during the restoration attempt. Proper recording of results is particularly important if conclusions reached are to be presented as courtroom testimony. The procedure recommended here is based upon suggestions of C. W. Cook of the Colorado Bureau of Investigation and A. R. Paholke of the Chicago Police Department (4-6).

As a restoration attempt proceeds, it is possible for a portion of a number to appear and then disappear with continued treatment. Thus, all observations must be promptly recorded. The following procedure should be followed:

Step 1. Establish the number of digits believed obliterated
and mark that number of spaces on paper.

——— ——— ——— ——— ——— ———

Step 2. Record numbers or parts of numbers as soon as
they are visible.

1 ∪ ∠ 5 ___ ꓛ
——— ——— ——— ——— ——— ———

Step 3. Continue treatment until no further recovery is
possible.

1 ꓳ 2 5 ___ ꓛ
——— ——— ——— ——— ——— ———

Step 4. Below each space, record all possible interpreta-
tions of what the number is. Take care to show the style
of numbers recovered.

1 ꓳ 2 5 ___ ꓛ
——— ——— ——— ——— ——— ———

(1)   0   2   5   (?)   (3)
(4)   (5)
                                               (6)
                                               (8)

When the specimen is implicated in a serious crime, a photographic
record of the restoration may be desired. If the recovered number is de-
pressed below the surface, photographic quality often can be improved by
lightly polishing the specimen or dusting with powder and wiping off the
excess.

After completion of restoration work, preservation of the specimen surface may be a concern. A surface that has been polished is subject to corrosion, particularly if it has been treated with etching chemicals. To avoid corrosion the specimen should be thoroughly rinsed with water followed by acetone. It is advisable to apply a protective layer of oil or clear lacquer.

Restoration from Specimen Reverse Side

If a specimen is made from thin metal stock, deformation from stamping can extend completely through the piece and reach its back side. Figure 8-3 shows an extreme case in which a serial number can be read directly on the back side of a stamped aluminum vehicle identification tag with no restoration work having been done. Even if deformation is not extensive enough to allow direct reading of the number, recovery may be possible by application of a conventional restoration procedure to the back side of the specimen. Such an approach would be advantageous if the amount of polishing required on a fairly smooth back side were less than that on a roughly obliterated front side.

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2. "Nondestructive Rapid Identification of Metals and Alloys by Spot Test," ASTM Special Technical Publication 550, American Society for Testing and materials, Philadelphia, 1974.

3. Krema, Vaclav, The Identification and Registration of Firearms, Charles C. Thomas, Publisher, Springfield, Ill., 1971.

4. Cook, Claude W., The Restoration of Obliterated Stamped Markings on Metal, Colorado Bureau of Investigation, Denver, Colo. No date.

5. Cook, Claude W., "The Restoration of Obliterated Stamped Markings on Metal." Presented at the Conference of the Association of Firearm and Toolmark Examiners, San Diego, Calif., April, 1975.

6. Paholke, Arthur R, Criminalistics Division, Chicago Police Department, personal communication, 1975.

Table 8-1

Nitric Acid Spot Test Scheme for
Identification of Metals and Alloys

	Reaction during first minute	Appearance after five minutes	Appearance after ten minutes and water rinse
Magnetic metals			
Alloy steel AISI 4140	Black ring slowly forms	Clear drop with black ring	Gray spot
Cast iron ASTM 4040	Black ring	Black-green drop	Black-brown spot
Low-carbon steel AISI 1116	Effervesence and brown drop	No change	Brown ring
Medium-carbon steel AISI 1137	Gray ring	Clear drop with brown-gray	Gray ring
Nickel plate	Yellow drop	Blue-green drop	Gray spot
Tool steel AISI O1	Black ring slowly forms	Brown-green drop and mild effervesence	Black-brown spot
Nonmagnetic metals			
Aluminum alloy AA 6063	None	No reaction	No spot
Aluminum/copper alloy AA 2014	None	No reaction	No spot
Aluminum/silicon alloy AA 333	None	No reaction	No spot
Brass ASTM B-16	Effervesence and green drop	No change	Clean spot
Lead	None	Dulled surface	No spot
Stainless steel Type 301	None	No reaction	No spot
Tin	Effervesence and yellow-white drop	White drop	Light gray spot

Table 8-1 (Cont.)

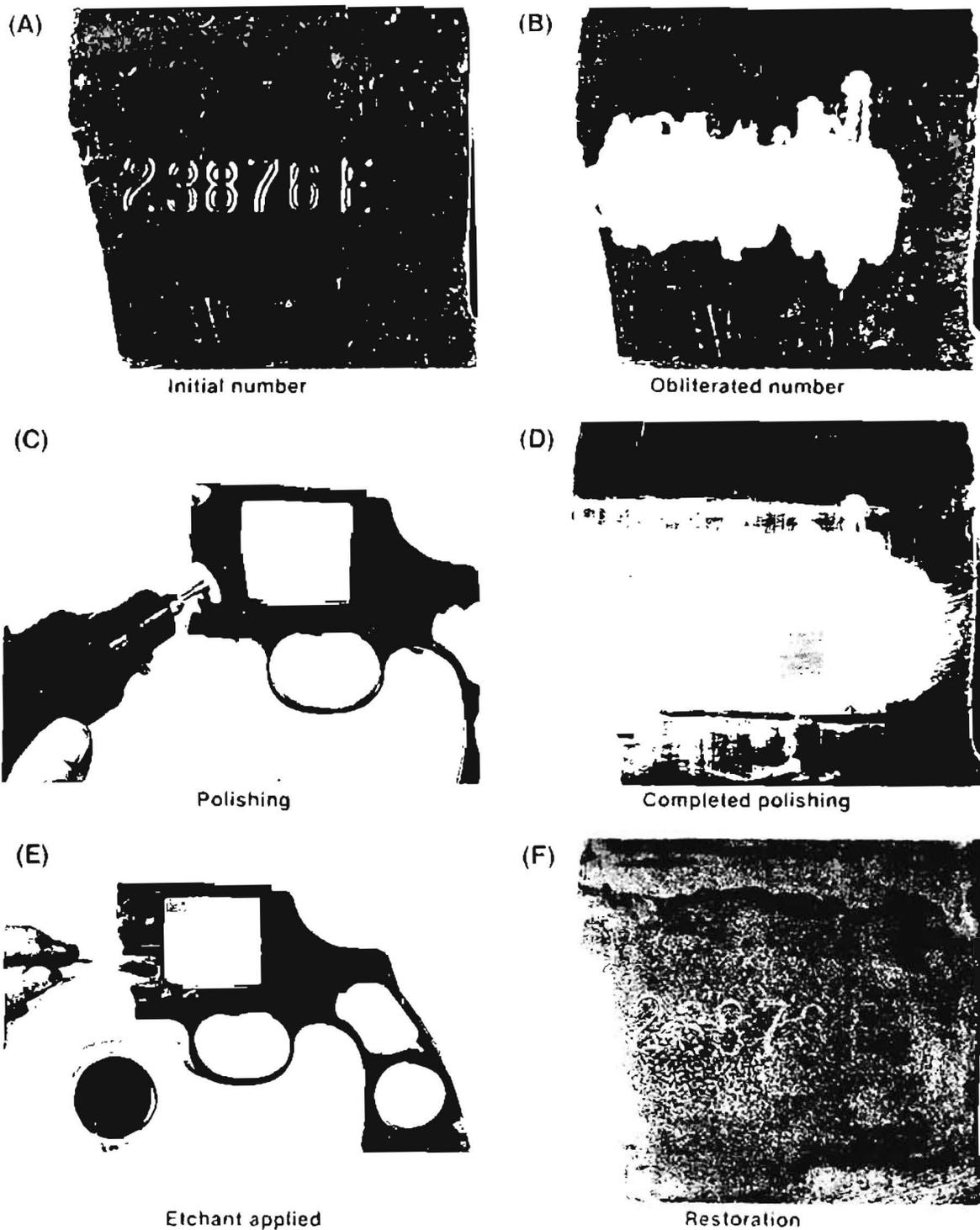|	Reaction during first minute	Appearance after five minutes	Appearance after ten minutes and water rinse
Zinc alloy ASTM AG-40A	Effervesence and black drop	Black drop	Dark gray spot

Figure 8-1. Sequence of steps in the chemical method restoration of serial number on Colt Model-I revolver frame

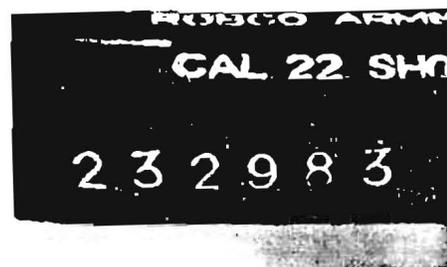|		
Benan	Charter Arms	Colt
Garcia	High Standard	Interarms
M.A.B.	RG	Rohm
Rosco Arms	Rossi	Titan

Figure 8-2. Styles of numerals used for selected handgun serial numbers.

(A)

(B)

Figure 8-3. Vehicle identification number on aluminum tag. (A) From front and (B) Reverse side.