16

Metallographic Etching of Aluminium and Its Alloys for Restoration of Obliterated Marks in Forensic Science Practice and Investigations

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1. Introduction

1.1 The problem

A problem of common occurrence in forensic science is the restoration of obliterated serial numbers on the chassis and engine of stolen motor vehicles, firearms, jewellery, valuable tools, and machinery (Nickols, 1956; Wolfer & Lee, 1960; Jackson, 1962; Cunliffe & Piazaa, 1980; De Forest & Gaensslen, 1983; Schaefer, 1987; O'Hara & O'Hara, 1994; Moenssens *et al* (1995); Heard, 1997; Petterd, 2000; Lyle, 2004; Katterwe, 2006; Seigel, 2007; Mozayani & Noziglia, 2006; Jackson *et al* (2008); Levin, 2010). Serial numbers or other markings, which are unique to that particular item, are usually marked on the above metal surfaces during the manufacturing process. Criminals alter or obliterate these identification marks during thefts or other illegal uses in order to prevent their identity. On many occasions a fraudulent number would be introduced after removing the original one. In abandoned vehicles all serial numbers are verified in order to detect alterations in the identity of the vehicle (Svensson *et al*, 1981). Sometimes the serial numbers on firearms are removed more professionally making it hard to distinguish whether the numbers are original or not (Shoshani *et al*, 2001). Restoration of the original obliterated numbers provides important forensic evidence in order to return the items to the owner and also to follow up the criminal proceedings against the perpetrator. In cases involving firearms the recovered numbers provide an important investigative lead. The history and the ownership of the firearm can be obtained only from the serial number. In forensic practice a few experimental techniques are being used successfully to restore the obliterated serial numbers on the above surfaces. The methods include: *chemical etching, heat treatment, magnetic particle, ultrasonic cavitation and relief polishing* (Nickols, 1956; Polk & Giessen, 1989; Maxwell, 1993; Heard, 1997; Hogan *et al*, 2006; Katterwe, 2006). Nondestructive methods such as hardness testing, magnetic imaging, x-ray imaging, eddy current, infrared imaging, scanning acoustic microscopy, electron channeling contrast and thermal wave imaging are also proposed (Hogan *et al*, 2005; Katterwe, 2006; Klees, 2009). However, chemical etching is most popular because it is sensitive, easy to apply on any size and also kind of object and reproducible. Etching can be applied on all metal surfaces including precious ones (Crowe & Smith, 2005).

This chapter presents background information on serial number restoration and etching techniques applied to recover the obliterated markings on aluminium and especially two of

its important alloys, Al-Zn-Mg-Cu and Al-Si. The etching results arising from some of these surfaces are illustrated. For the sake of completeness, some brief notes on the classical recovery of obliterated marks on iron and steel surfaces and use of methods other than chemical etching are also added.

1.2 Serial number marking methods

The common methods of marking objects include: *stamping, pin stamping, type wheel marking, engraving, laser etching* and *embossing* (Collins, 1999; Petterd, 2000; Katterwe, 2006). The serial numbering on motor vehicles and firearms is generally performed by *stamping*. In this process the steel die bearing the digit is struck with a single stroke manually or using a pressing machine. This causes an indented character. The amount of pressure applied per character to achieve a certain depth depends on the hardness of the metal surface. In *pin stamping* small pins are used to form individual dots on the substrate. Like in stamping, metal is being compressed beyond its elastic limit and a permanent impression is created. The impact pin is made of tungsten carbide and is driven rapidly toward the surface to be marked. Pin stamping is applied to mark vehicle identification number (VIN). *Type wheel markings* are made in a hydraulic press with type wheels controlled by computers. This type of marking is used by some automobile manufacturers with VIN. *Engraving* is a chip cutting operation where the substrate is cut away by a tiny spinning head leaving the marks. *Laser etching* uses an intense laser beam operated by a computer controlled system. Quality, depth and precision of laser marking are controlled by laser frequency, scan speed and Q-switching. Automobile components and modern firearms are currently being marked by laser. The characters are usually shallow and they are visible to the eye only because the heat of the laser changes the appearance of the metal inside each character (Collins, 1999). *Embossing* is similar to die stamping, but used on thin plates. The die is pushed onto the plate from behind producing a raised appearance of the marks.

1.3 Stress and deformation on the metal

Metallurgical aspects of serial number recovery are described in (Turley, 1987; Polk & Giessen, 1989; Collins, 1999; Petterd, 2000; Katterwe, 2006; Hogan *et al*, 2006). The atoms within metals of interest have a three dimensionally periodic crystalline arrangement within local regions. These regions are called grains. Between the grains are interlocking regions known as grain boundaries. The strength of these boundaries relates directly to the strength of the metal surface and its ability to resist stress. When a number is stamped onto a metal surface using sufficient pressure, the metal is unable to resist it and a permanent change in shape takes place. The grains around and beneath the indented characters are deformed and the alignment of the crystal structure is altered. This deformation known as cold work will not only be confined to the crystals immediately below the character, but will extend to a considerable region below the impression. The deformation is carried deeper than the depth of the marking themselves. This can be referred to as zone of plastic strain. Thus, the stamping operation produces a visible indentation of the mark and a plastically deformed region underneath it extending to a considerable depth. Significantly, an imprint of the mark is latent below the visible indentation in the form of plastically deformed regions. In other words, the region of plastic strain carries information of the stamped number and it is crucial to number restoration in forensic investigations. Depth of this strain is dependent on the metal, the size and shape of the die and the force used to produce the indentation.

Metallographic Etching of Aluminium and Its Alloys for Restoration
of Obliterated Marks in Forensic Science Practice and Investigations

333

Experiments have demonstrated that in case of zinc the zone may run to depths of at least 20 times the depth of the deepest character (Kehl, 1949), while in steel the affected region may be six times as deep as the stamped mark (Thornton & Cashman, 1976). In cases of engraved marks on steel and aluminium surfaces the depths of recoverable deformations varied between 2 and 3 times the depths of engraving (Azlan *et al*, 2007; Izhar *et al* 2008; Bong & Kuppuswamy, 2010; Uli *et al*, 2010).

In cases of marks produced by pin stamping and engraving the depth of plastic deformation or strain is not as great as in stamping. Pin stamped and engraved characters, if obliterated, can be recovered but are more sensitive to the depth of erasure (Collins, 1999). Laser etching on the other hand has no deformation but produces heat affected zone (HAZ). The depth of this zone will depend on the intensity of the laser and scan speed.

The properties of the metals are known to change because of the altered crystalline structure of the metal during marking a number (Thornton & Cashman, 1976; Polk & Giessen, 1989; Hogan *et al*, 2006). The cold worked or deformed region has physical and chemical properties different from that of nondeformed metal. Important property changes are an increase in hardness and decrease in ductility and yield stress. In addition changes occur in the electrical resistivity, the magnetization behaviour of ferromagnetic alloys, the electronic work function and the chemical potential among others. Every one of these property changes is used advantageously to detecting the plastically deformed regions that are residually present after the visible indentations of serial numbers have been removed. For example, hardness differences are exploited in chemical etching, ultrasonic cavitation and relief polishing, while differences in magnetic properties are utilized in magnetic particle method. Heat treatment applicable to cast iron substrates relies on the residual stresses present below the stamped region (Kehl, 1949; Maxwell, 1988; Katterwe, 2006).

1.4 Methods used for the removal of the markings

Criminals have used the following methods for the removal of the serial numbers: *filing or grinding, peening, welding and drilling* (Cook, 1989; Heard, 1997; Petterd, 2000; Katterwe, 2006). During grinding or filing the marks are usually removed till the base of metal so that the number would no longer be visible. If the erasure of the serial number does not go deep enough to remove the underlying plastic zone, the obliterated marks may be restored by suitable methods. But, if the metal removal extends entirely into the plastic region, the numbers cannot be recovered. Sometimes the perpetrator after grinding the area will polish it and substitute a false number. This process introduces a new deformation overlying the original deformation.

Occasionally, there will be no removal of metal by grinding but the marks will be defaced by peening so that they will not be discernible. This alteration involves hammering the mark using centre punch or cold chisel. The degree of deformation caused by peening can vary. If the deformation caused by peening operation is localized or superficial recovery may be possible.

Drilling and welding of the area bearing numbers are also encountered. With drilling, the outline of the numbers may sometimes become visible by careful polishing of the area and examining it using a stereomicroscope under oblique lighting conditions. However, when the numbers are fused by welding, the recovery is almost impossible. Here the plastic strain carrying the original number would be relieved due to heating.

Markings may also be converted using dies of similar size and style. For example, the digit 6 can be changed into 8. A careful examination under magnification will reveal in such cases

discontinuity of the outlines, unevenness of stamping and additional marks (Massiah, 1976; Kuppuswamy & Senthilkumar, 2004).

2. Etching of metal surfaces for restoration of obliterated marks

Macroetching techniques are probably the most informative and widely used for quality control, failure analysis and in research studies in material science (Vander Voort, 1984). The mechanical inhomogeneity in a metal caused by stamping or other marking process will be determined by macroetching the obliterated surface and examining the marks revealed with the unaided eye or with the aid of a hand magnifier.

A wide variety of chemical etching reagents for iron and steel and common nonferrous metals and alloys are available in literature (Kehl, 1949; Nickols, 1959; Metallographic reagents for iron and steel, 1974; Massiah, 1976; Petzow, 1976; Vander Voort, 1984). Detailed etching procedures and time of etching are also compiled therein. Composition of the alloy will strongly influence the choice of a particular etchant.

Before the etching process begins, the item should be photographed showing details of the obliterated area. If necessary, the area may be dusted with fingerprint powder and lifted with tape. The area should be degreased using alcohol/acetone before any surface preparation.

2.1 Mechanism of etching

Etching is basically a controlled corrosion process resulting from electrolytic action between surface areas of different potential (Kehl, 1949; Vander Voort, 1984; Polk & Giessen, 1989). The etching solution does not act in a uniform manner on different faces of the crystals, or on different areas of the grain. The cold worked regions are known to be chemically more reactive, as the position of the deformed metal in the electromotive series is raised. Hence they will dissolve in an acid at a more rapid rate than the surrounding unaffected regions (regions not affected by stamping). The desired result is a local change in reflectivity of light that reveals the obliterated marks (Polk & Giessen, 1989; Thornton & Cashman, 1976). Thus the etching technique relies on the difference in the etching behaviour between the deformed and undeformed regions in order to produce the desired image contrast.

2.2 Surface preparation

Metallurgical literature devotes considerable attention to specimen preparation before etching (Kehl, 1949; Vander Voort, 1984; Vander Voort, 1986). The steps mainly include grinding (using coarse abrasives) and subsequent polishing of the metal surface (using fine abrasives) in order to study the microscopic structural characteristics. However in forensic practice the area of the engine/chassis number of a car, for example, may contain a lot of scratches and gouges caused during the obliteration of the number. A careful microscopic examination with illumination from low angle will sometimes reveal traces of the numbers against the scratches prior to any surface preparation (Matthews, 1962; Srinivasan & Thirunavukkarasu, 1996). The presence of the cutting marks, however, may interfere with the interpretation of the recovered marks during etching. Hence the obliterated surface should be made smoother by use of emery papers of different grades starting from coarser grades and finishing with finer ones (Polk & Giessen, 1989; Katterwe, 2006). Careful polishing producing smooth finish free from all grinding scratches is to be preferred. This is important as such scratches may affect the contrast of the recovered number. However care should be taken that more metal that includes the residual deformation zone is not removed during preparation stage.

Metallographic Etching of Aluminium and Its Alloys for Restoration
of Obliterated Marks in Forensic Science Practice and Investigations
335

Sometimes the obliterated serial number plate of a firearm has to be removed and examined from its back to reveal the negative impression caused by the stamping of the front side of the plate (Cooper, 2002).

2.3 Etching technique

Chemical etchants are used at room temperature either by swabbing the sample or immersing and gently agitating it (Kehl, 1949; Nickols, 1959; Massiah, 1976; Petzow, 1976; Vander Voort, 1984; Katterwe, 2006). The surface is swabbed by use of cotton-tipped applicator. The cotton should be dipped regularly in fresh etchant and applied until the desired contrast is obtained. The swab is moved slowly back and forth across the surface with gentle pressure. The surface is observed closely for the appearance of any marks. Swabbing is mostly preferred in number restoration, as it provides control over etching technique. Immersion is preferred in cases where restoration is slow to developing. In such situations a small surround of plasticine is built around the obliterated area and the reagent is filled within (Nichols, 1959; Petterd, 2000). The reagent is removed at intervals and replenished. Control of the etching time is important as the obliterated numbers would appear and disappear during the etching. While some restorations appear within minutes of reagent application, others would take about an hour or more. Under-etching fails to reveal the obliterated marks, while over-etching destroys the recovered marks. As etching time and temperature are closely related, it is desirable to do etching at room temperature.

2.4 Photography of the recovered marks

Photography is the best method for permanent record of the restored marks. Photographic methods are similar to those used for recording latent fingerprints and also documents dealing with obliterations. Recovered marks are inherently very faint and the contrast with the background would be usually poor. They can further be seen only at certain angles of illumination. The examiner should be familiar with close-up and macrophotographic techniques. In cases of firearms where the specimen is relatively small, macrophotography of the marks can be carried out in the photo-copying stand using appropriate illumination. However, when dealing with engine and chassis of cars the camera has to be set up on an adjustable tripod. Several exposures are to be made by shifting the light source so that it illuminates the surface from different directions. Black and white photography using high contrast film may also help to bring out the faint outlines of the number. Light reflected by a mirror may be advantageously used to record the faintly recovered digits. A combination of flash and available light sources is also recommended (Katterwe, 2006). Current digital photographic techniques provide greater advantage offering contrast enhancement software procedures. Sometimes the area carrying the recovered marks was dusted with fluorescence finger printing powders (Lightning Redwop and Lightning Greenwop) and a piece of clean cellophane tape was used to lift the marks. The lifted tape, when viewed through a 550 nm barrier filter using an alternate light source at 450 nm bandwidth, produced excellent contrast of the marks (Nalini & Hemalatha, 2003).

2.5 Specimen storage after etching experiments

When the etching examination is completed the area of the chassis, engine or frame of firearm should be thoroughly washed in a stream of distilled water and acid neutralized using ammonia (Nicholls, 1956; Massiah, 1976). The surface should be dried and the area is covered with grease or shellac to prevent oxidation or further etching. When cars and

firearms are to be stored for long periods of time during criminal proceedings, they must be protected from atmospheric corrosion.

3. Macroetchants for revealing strain patterns in steel

In metallurgical investigations visualization of macrostructure is carried out by macroetching (Kehl, 1949; Petzow, 1976; Vander Voort, 1984). For study of steels strong acids were used initially for "deep" etching. Later copper-containing macroetchants were used for studying primary structure. These procedures were quite unsuitable to develop the plastically deformed regions in order to reveal the obliterated marks. It was Fry in 1921 who published macroetchants for revealing strain patterns on iron and steel (Vander Voort, 1984). His reagent is very popular even today and has many variations. The most popular composition is cupric chloride 90 g, hydrochloric acid (conc.) 120 ml and water 100 ml, which is used exclusively to restore marks on the chassis and engine of motor vehicles and frames of firearms. This solution contains considerable HCl that keeps the free copper from depositing on the sample during etching (Vander Voort, 1984). Applying this reagent many successful recoveries are reported in literature (Nickolls, 1956; Matthews, 1962; Wilson, 1979; Heard, 1997; Kuppuswamy & Senthilkumar, 2004; Warlow, 2004; Wightman & Matthew, 2008; Siaw & Kuppuswamy, 2009). (Turley, 1987) used this reagent to study the relationship between the depth of the stamp mark and the depth to which stamp marks could be recovered on plain carbon steels ranging in carbon content from 0.04 to 0.52% C. He noted that the depth of restoration increased with increased depth of stamp mark. However, the increase was nonlinear. He also found that etching with this reagent was superior to magnetic techniques for old stamp marks and also to recover the marks obliterated by overpunching. More recently (Azlan *et al*, 2007) found one variation of Fry's reagent with composition of 5 g $CuSO_4$, 60 ml water, 30 ml conc. ammonium hydroxide and 60 ml water to be most sensitive to low carbon steel surfaces, while (Siaw & Kuppuswamy, 2009) noted that the most popular one (90 g $CuCl_2$, 120 ml HCl (conc.) and 100 ml H_2O) to be quite effective on medium carbon steel surfaces used in the chassis of cars. Recently (Wightman & Matthew, 2008) reported that even though Fry's reagent was an effective etch for recovering erased marks in steel, ageing of the reagent gave a decrease in performance and took longer time to etch. The same authors (Wightman & Matthew, 2008a) have proposed thixotropic etching paste of Fry's reagent using alumina which provided more controlled chemical attack with obliterated surfaces and found that the recovery was better. Thus Fry's composition is contributing enormously to the success of serial number restoration for steel surfaces for many decades. (Heard, 1997) remarked that although Fry's reagent is the best for steel, it can also be used, with care, on any other material. (Massiah, 1976) also reported that usual reagents for aluminium when used along with Fry's reagent would be more effective.

4. Restoration techniques for aluminium and its alloys

Etching techniques for obliterated stamped marks for iron and steel using Fry's reagent have been described in detail in forensic literature more than any other metal because of the greater use of these in automotive engines and chassis and also firearms. There has been frequent tampering of serial numbers on these items. An important development which is of concern to forensic examiners is in the use of aluminium alloys in motor vehicle components and more especially in frames of firearms. Aluminium is a relatively weak material. For

applications requiring greater mechanical strength it is alloyed with metals such as copper, magnesium, manganese and zinc, usually in combination with two or more of these elements together with iron and silicon (King, 1987). Aluminium alloys are widely utilized in various structures and components because of their good strength and low density. In addition many automobile parts have been manufactured with Al alloys instead of steel materials to reduce the weight of the car (Okayasu *et al*, 2008). In addition many firearms like Ruger pistol, Czechoslavakian P-01 pistol, S & W Special Airweight Airlite revolver and Brazilian pistols use aluminum alloy frames. Ergal, a hard aluminium alloy (AA 7075) is also used for manufacturing firearms (Bond, 2008).

4.1 Recovery of stamped marks by chemical etching

Many etching reagents are recommended in metallurgical literature for aluminium alloys (Mondolfo, 1943; Kehl, 1949; Nickols, 1956; Massiah, 1976; Petzow, 1976; Vander Voort, 1984). Among them sodium hydroxide is one of the reagents most commonly employed in a variety of concentrations. All these reagents are mainly useful in material science investigations for the identification of the constituents of the alloy or the crystals themselves. However, only a few etchants among them will be able to reveal the plastic deformation zones carrying original markings. Recovery of marks on aluminium surfaces unlike steel is difficult. There has been no popular reagent to etch aluminium surfaces as we find in Fry's composition for steel. The standard reagents used in etching on iron and other metal surfaces provide too vigorous reaction with aluminium resulting in pitted metal and blurred results (Chisum, 1966). There have been a few attempts in the past to identify suitable etchants. These works are summed up below.

(Nickolls, 1956) recommended the use of Villela's solution comprising 2 parts HF, 1 part HNO_3 and 3 to 4 parts glycerin, as it responded to treatment to all aluminium alloys. For castings made with high silicon alloys he suggested to give a quick rub with Hume-Rothery's reagent: 200 g $CuCl_2$, 5 ml HCl, and 1 l H_2O. He also illustrated successful restoration on an aluminium crank case developed with this procedure. Villela's reagent should not be stored in glass bottles. (Masssiah, 1976) compiled a list of etchants for different metal surfaces and his work is a useful reference for number restoration procedures. He advocated use of the reagent comprising 200 ml (conc.) HCl and 200 ml distilled water. This mixture was saturated with cupric ammonium chloride. He also suggested diluting the above reagent for optimal results on different aluminium alloys. (Chisum, 1966) used the following reagents: (i) 1N NaOH and (ii) 0.1M $HgCl_2$ in 0.1N HCl after polishing by sanding. A preliminary wash of the surface with 1N NaOH gave more even coating of Hg. After removing this wash, a solution of 0.1M $HgCl_2$ was applied with a cotton swab. The numbers appeared after the aluminium oxide mass was blown off. The mercury coating catalyzed the oxidation of aluminium and the number became visible because plastically deformed regions oxidized faster than the surrounding metal (Polk & Giessen, 1989). The working time excluding of polishing was 20 min. Brown (2001) noted that the Ruger P-Series aluminium alloy framed pistol was found to be most difficult to restore erased serial numbers. He used acidic ferric chloride chemical reagent (25 g $FeCl_3$, 25 ml HCl, and 100 ml H_2O) after polishing the metal surface using water and 600-grit sand paper. He used a modified application of the etchant. The reagent was added drop wise using saturated cotton tipped swab and the tip of the swab was lightly pressed directly to the frame and removed. No wiping of the swab across the frame was performed. The reagent effervesced for 1 to 2 min and was removed by spraying with distilled water. After a few

applications of the above procedure for 10 min the number appeared as darker "ghostly" lettering. The contrast however was extremely poor and ring lighting and oblique lighting did not improve it. (Klees, 2002) reported a case in which the serial number and barcode of a Smith & Wesson pistol was obliterated by abrading. The serial number area of the pistol consisted of nonferrous metal pad surrounded by a polymer skin. This metal pad was laser etched with 3/32"alphanumeric characters. He used a chemical etching reagent with a low corrosive rate with the nonferrous metallic media to fully restore the serial number and partially restored barcode. He however did not mention the composition of the reagent he used. Importantly, he described procedures on how to decrypt manually the partially recovered barcode. (Peeler *et al*, 2008) developed stamped marks on Al motor cycle frames that involved the swabbing of 60% HCl for about 1 h or until "drag" was felt on the swab, then flushing with water to remove all etchant and continuing etching with 40% NaOH until no further darkening of the surface was observed. The cycle was repeated if necessary. The etching took as nearly as 6 h for acid swabbing followed by alkali swabbing for about 1 h. The time varied according to the substrate. The materials they tested were Al-Si and Al-Mg alloys. They reported that many numbers were able to be recovered without the use of the alkali; however the use of the alkali after acid swabbing enhanced the contrast between stamped and unstamped areas. They also successfully used the above method for restoration of 9 motorcycle frames involved in criminal cases.

It is known that the strain patterns in most metals can be revealed by annealing the specimen after deformation so as to obtain recrystallization. In the region that receives critical amount of strain, generally 5 to 8%, grain growth is more rapid (Vander Voort, 1984). This area is visualized more clearly on etching. (Okayasu *et al,* 2008) used this principle to reveal the plastic deformation zone in aluminium alloys such as 2017 duralumin. A severe plastic deformation was imposed by applying a compressive force of 5 kN at a rate 1 mm/min onto the specimen. The deformed samples were heated to 673 K for 3 h. The surface was polished to mirror finish and etched for 1 min using Dix-Keller reagent consisting of 4 ml HF, 6 ml HCl, 8 ml HNO_3, 80 ml H_2O and 50 ml ethyl alcohol. After etching the plastic deformation zone was darkly etched, but the undeformed zone lightly etched. The deformation zone was in agreement with the microhardness measurements.

Although the etching methods described above are the most important and widely used procedures for revealing the obliterated marks on aluminium surfaces, it is sometimes possible to restore the obliterated numbers by methods other than etching.

4.2 Recovery of embossed marks using x-ray radiography

X-ray radiograph is known to detect internal variations and defects in metals and it provides permanent records in the form of photographic films and image files (Jeon *et al,* 2009). The use of X-rays to recover obliterated marks has not been successful to visualize the minute damaged areas left after the removal of the number (Petterd, 2000; Katterwe, 2006). However, the method has been successfully used to locate serial numbers that have been hidden with paint or by welding another piece of metal on top of the original (Nickolls, 1956; Petterd, 2000; Katterwe, 2006). (Jeon *et al,* 2009) investigated the forged embossed numbers on car license plates of 1-mm thick and 335x170 mm aluminium sheet. In this case the criminals had obliterated the original number by painting the plate and flattened it by hammering. They then embossed a new number over the flattened one and painted it. However, when the plate was exposed to x-rays with 30 kV/mm and 20 mA for 2 min and

Metallographic Etching of Aluminium and Its Alloys for Restoration
of Obliterated Marks in Forensic Science Practice and Investigations
339

the resultant radiographs were treated with image processing programme the original numbers appeared quite distinctly.

4.3 Recovery of laser etched marks—alternate approaches
4.3.1 Relief polishing
Relief polishing works successfully where it is known that an alloy is composed of relatively hard and soft structural constituents. By hand polishing it is possible to abrade the softer constituent at a greater rate than the harder one, thus leaving the harder phase in bold relief at the end of the polishing operation. When such polished specimen is examined microscopically using oblique illumination difference between structural details will be seen (Kehl, 1949; Katterwe, 2006). Following this principle (da Silva & dos Santos, 2008) restored the partially obliterated laser engraved serial number on aluminium-alloy frames of Brazilian pistols. It is known that laser engraving creates heat affected zone of 2-25 µm which has greater hardness than the rest of the metal. The obliterated area was polished using abrasive papers 600, 1200 and water. The images were captured in a stereomicroscope while the polishing was on. The scratches caused during obliteration of the number were removed at a faster rate than the original marks produced by the laser and the original numbers became visible. This was due to the difference of hardness in the regions where the number was written by laser and the remaining region of the frame. The difference in removal rates allowed the original digits or fragments of them to appear. However, these authors noted that relief polishing cannot be applied to frames which are damaged or carrying deep scratches. Such polishing removed both the digits and the scratches.

4.3.2 Adobe Photoshop
(Klees, 2009) recently used Adobe Photoshop in one real forensic investigation. Here, the laser-etched bar code and serial numbers of a Smith & Wesson pistol were partially obliterated. A digital photograph of the obliterated area was produced and enhanced for brightness and contrast using Adobe Photoshop. This procedure increased the visibility of the partial characters and bars. Using the magnifier and Line tool, the obliterated portion of the bars were redrawn and scanned with a barcode reader to detect the original marks.

4.4 Restoration of obliterated mechanically engraved marks—recent findings
The present author along with Izhar and Rahman (Izhar *et al*, 2008), Bong (Bong & Kuppuswamy, 2010), and Norjaidi and Firdaus (Uli *et al*, 2010) reviewed the sensitivity and efficacy of some common etchants on Al (99% purity), Al-Zn-Mg-Cu and Al-Si alloys. High strength Al-Zn-Mg-Cu alloy finds applications in automobile components and frames of firearms, while Al-Si alloy is used as motor vehicle engines. The chemical composition of the aluminium specimens they investigated is given in **Table 1.**

Specimen	Cu	Mg	Si	Fe	Mn	Zn	Pb	Ni	Zr
Al (99% purity)	0.05	0.51	0.360	0.250	0.017	0.008	--	--	--
Al-Zn-Mg-Cu alloy	1.6	2.4	< 0.12 (Si+Fe)		--	6.3	--	--	0.14
Al-Si alloy	2.18	0.20	10.76	0.84	0.18	0.75	0.054	0.076	--

Table 1. Chemical composition of the aluminium specimens in % weight.

The experimental details and the results are summed up below.

Every one of the aluminium specimens (refer **Table 1**) was cut into small plates and marked with some characters by use of a computer controlled engraving machine "Gravograph". The machine made reproducible marks. The depths of engravings were between 0.02 to 0.03 mm (the difference in thickness before and after erasure of the marks). Engraved marks on the specimen plates were obliterated by grinding the marks (using sand papers of different grades - P80, P150 and finished with fine grade P320) to different depth levels upto and (0.01, 0.02, 0.03, 0.04 mm etc) below the bottom of the engraving depth. Several of such plates erased to different depth levels were prepared.

Further, engraved marks on some plates were obliterated by (i) peening, using centre punch till the marks were no longer discernible, and (ii) grinding the original marks and engraving a new mark over the erased area. The obliteration methods were comparable to those encountered in real forensic cases.

Eight metallographic etchants of which some contained NaOH in different concentrations alone or together with an acid were used. The reagent composition and the references are given in **Table 2.**

Serial number	Reagent Composition	Reference(s)
1	1. Hydrochloric acid 60% 2. Sodium hydroxide 40%	(Peeler *et al*, 2008) (Izhar *et al*, 2008)
2	10% aq. phosphoric acid	Mondolfo, 1943 Petzow, 1976 Vander Voort, 1984
3	1. 10% Sodium hydroxide 2. 10% Nitric acid	Petterd, 2000
4	1. Sodium hydroxide 10 g 2. Water 90 ml	Mondolfo, 1943 Kehl, 1949 Petzow, 1976 Vander Voort, 1984 Petterd, 2000 (Dong *et al*, 2005) Katterwe, 2006
5	Acid ferric chloride 1. Ferric chloride 25 g 2. Hydrochloric acid 25 ml 3. Water 100 ml	(De Forest *et al*, 1983) Brown, 2001
6	1. 1N sodium hydroxide (1 mol/l) 2. 0.1M mercuric chloride aq. (0. 1 mol /l) in 0.1N Hydrochloric acid (0.1 mol/l)	Chisum, 1966 (Polk & Giessen, 1989) Rowe, 2003
7	Hume -Rothery reagent 1. Cupric chloride 15 g in 100 ml of water 2. 50% HNO_3	Mondolfo, 1943 Vander Voort, 1984
8	Nital* 1. 1 part Nitric acid 2. 9 parts Ethanol	(Cunliffe & Piazza, 1980)

Table 2. Etching reagents used to restore erased engraved marks on aluminium and its alloy surfaces.

*Nital solution is a highly flammable liquid and concentrations greater than 5% should never be stored, as at 10 % it is comparable to rocket fuel in its volatility (Vander Voort, 1984).

Metallographic Etching of Aluminium and Its Alloys for Restoration
of Obliterated Marks in Forensic Science Practice and Investigations

341

No additional polishing was carried out before etching as the surfaces were previously smoothed during the erasure of the marks. Further polishing had the danger of removing the plastic zone which was of the order of micrometers in cases of engraving.

The specimen surfaces erased to different depths by grinding were etched with every one of the reagents given in **Table 2**. The reagents were applied either by swabbing or immersion (10% aq. phosphoric acid reagent only) and the recovery was closely monitored. The most efficacious reagent was determined to be the one which restored the marks on the plate erased to maximum depth and also that provided good contrast of the recovered marks with the background.

The selected reagent was then tested to recover marks on plates obliterated by peening and overengraving.

Among the eight common metallographic reagents tested, reagents 4 to 8 (in **Table 2**) did not give the desired contrast and sensitivity (see for details Izhar *et al*, 2008; Bong & Kuppuswamy, 2010; & Uli *et al*, 2010) and hence were not suitable. The reagents 1 to 3 produced superior results both in terms of contrast and sensitivity for etching specific aluminium surfaces.

The reagent 1, 60% HCl and 40% NaOH on alternate application on *aluminium (99% purity)* (Izhar *et al*, 2008) and *Al-Zn-Mg-Cu alloy* surfaces (Bong & Kuppuswamy, 2010) were quite successful for recovering the marks obliterated by erasure, overengraving and peening. These are seen in Figs. 1 to 6. The success of alternate application of acid and alkali to
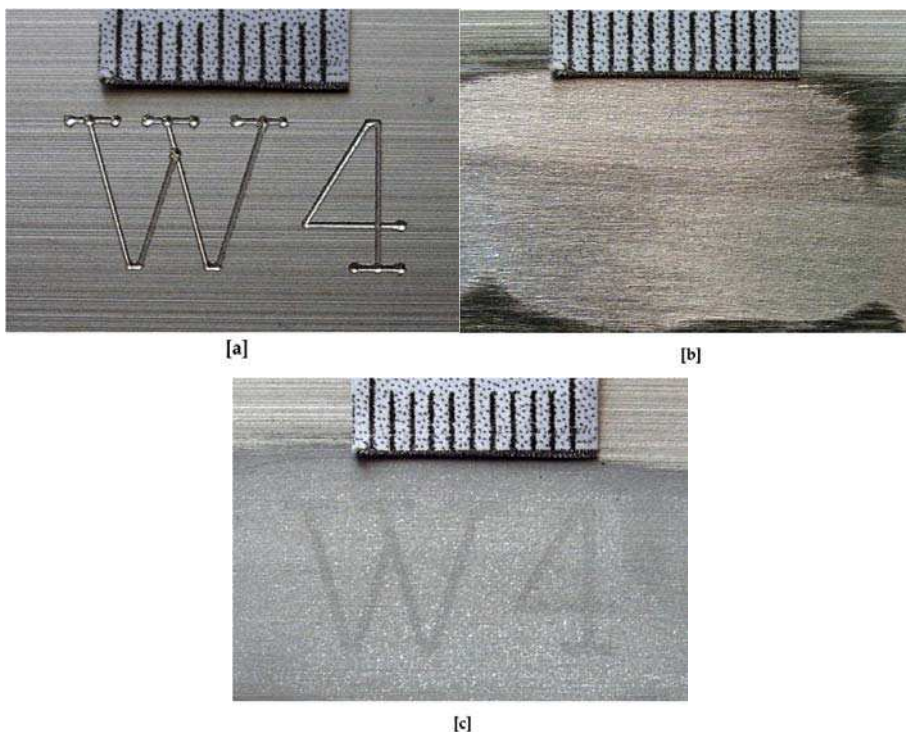

Fig. 1. a) The original engraved marks "W4" on Al (99% purity). b) Surface after grinding of the marks up to the depth of engraving (0.02 mm). c) The recovered number "W4" after etching process. Etching reagent: Alternating swabbing of 60% HCl and 40% NaOH. Etching time: 28 min.

Fig. 2. a) Original engraved marks MO3 on Al (99% purity) surface were erased (hence not seen) and a new marks W98 were engraved over the erased area. b) The original marks MO3 were restored in the presence of the new marks W98. Note partial overlapping of the original digits "0" and "3" respectively with the new ones "9" and "8". Etching reagent: Alternating swabbing of 60% HCl and 40% NaOH. Etching time: 20 min.
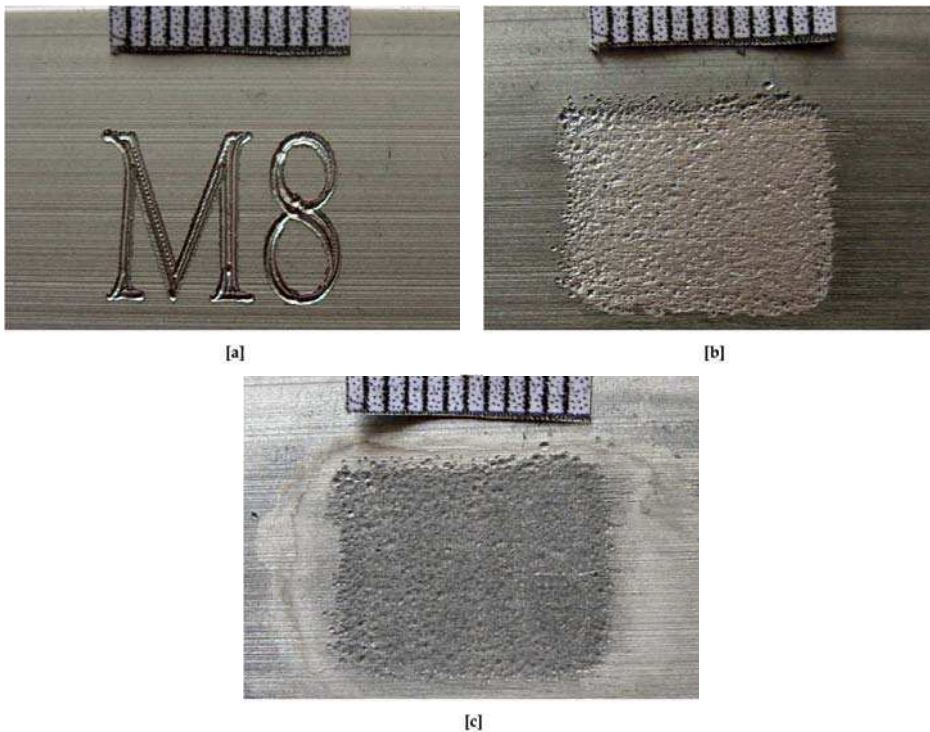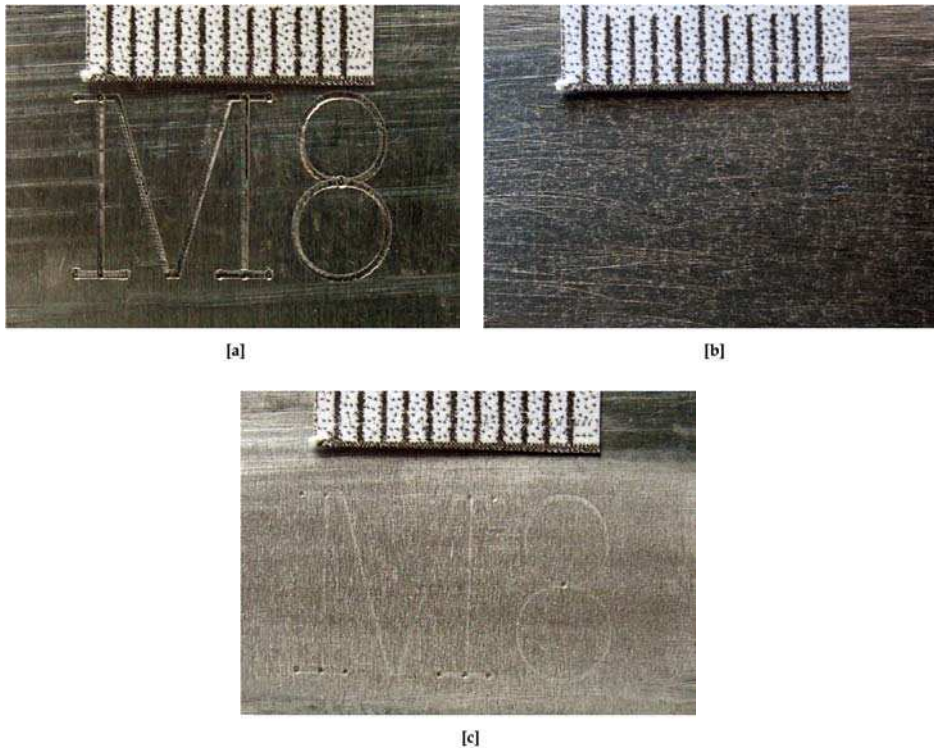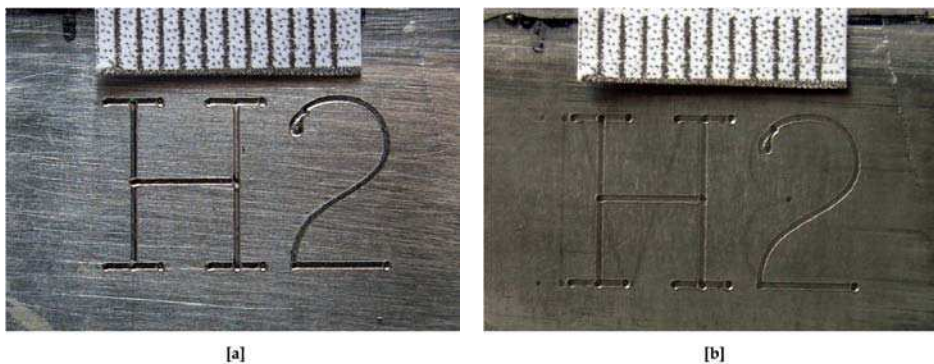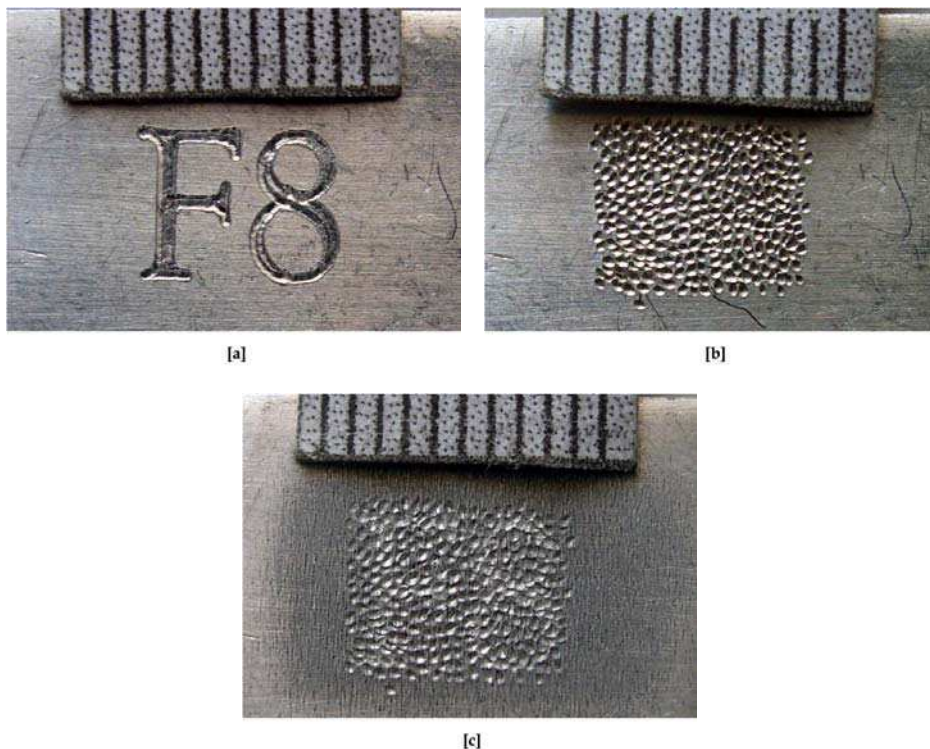

Fig. 3. a) The original engraved marks M8 on Al (99% purity). b) The marks were obliterated by peening using centre punch. c) See the outline of the recovered marks M8. Etching reagent: Alternating swabbing of 60% HCl and 40% NaOH. Etching time: 20 min.

Fig. 4. a) The original engraved marks "M8" on Al-Zn-Mg-Cu alloy surface. b) Surface after grinding of the marks up to the depth of engraving (0.02 mm). c) The recovered number "M8" after etching process. Etching reagent: Alternating swabbing of 60% HCl and 40% NaOH. Etching time: 16 min.

Fig. 5. a) Original engraved marks "M8" on Al-Zn-Mg-Cu alloy surface were erased (hence not seen) and in their place a new marks "H2" were engraved. (b) The original marks "M8" were restored faintly in the presence of "H2". Etching reagent: Alternating swabbing of 60% HCl and 40% NaOH. Etching time: 32 min.

Fig. 6. a) The original engraved marks "F8" on Al-Zn-Mg-Cu alloy surface. b) The marks were obliterated by peening using centre punch. c) The outline of the obliterated marks "F8" were recovered quite distinctly with good contrast. Etching reagent: Alternating swabbing of 60% HCl and 40% NaOH. Etching time: 12 min.

restoring the marks was attributed to the amphoteric nature of aluminium (Massiah, 1976; Dong *et al*, 2005). Aluminium could be etched both with acid and alkali. A specific percentage of each of these on alternate swabbing produced the desired results.

For *Al-Zn-Mg-Cu alloys* (Bong & Kuppuswamy, 2010) another application, 10 % aq. phosphoric acid by immersion for extended hours recovered the marks with excellent contrast. The results provided by this reagent are shown in Figs. 7 to 9. Phosphoric acid worked well on Al-Zn-Mg-Cu alloy because the reagent provided good etching on Mg_5Al_8. Further it was reported that this reagent showed strain in Al-Mg base alloys (Petzow, 1976; Vander Voort, 1984).

Comparing the etching response of phosphoric acid with that of alternating 60% HCl and 40% NaOH, it was noted that the former was able to provide better contrast and sensitivity, although the erased surface was to be immersed for long hours. It is recommended that when using this reagent the obliterated surface is immersed at least for 1 h and a closer watch is followed thereafter; otherwise the marks disappeared when the immersion was prolonged. More work is needed to utilize completely the potentiality of this etchant for high strength aluminium alloys.

Metallographic Etching of Aluminium and Its Alloys for Restoration
of Obliterated Marks in Forensic Science Practice and Investigations
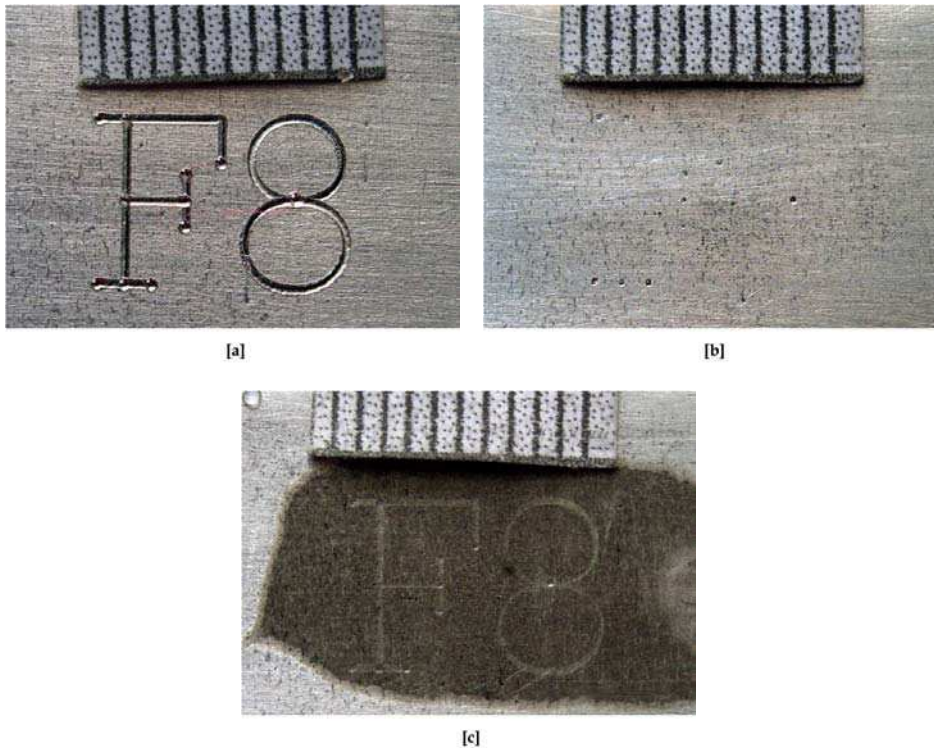345

Fig. 7. a) The original engraved marks "F8" on Al-Zn-Mg-Cu alloy surface. b) Surface after grinding of the marks up to the depth of engraving (0.02 mm). c) The recovered number "F8" after etching process. Etching reagent: 10% aq. phosphoric acid by immersion. Etching time: 6 h.
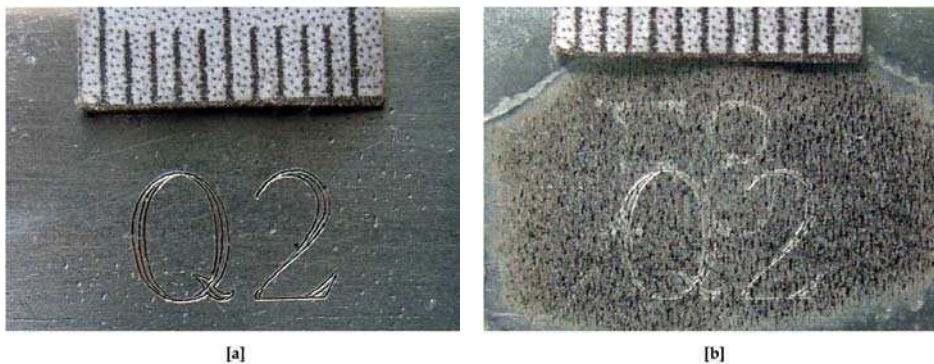

Fig. 8. a) Original engraved marks "F8" were erased on Al-Zn-Mg-Cu alloy surface (hence not seen) and in their place a new marks "Q2" were engraved. (b) The original marks "F8" were restored in the presence of "Q2". Etching reagent: 10% aq. phosphoric acid by immersion. Etching time: 8 h.
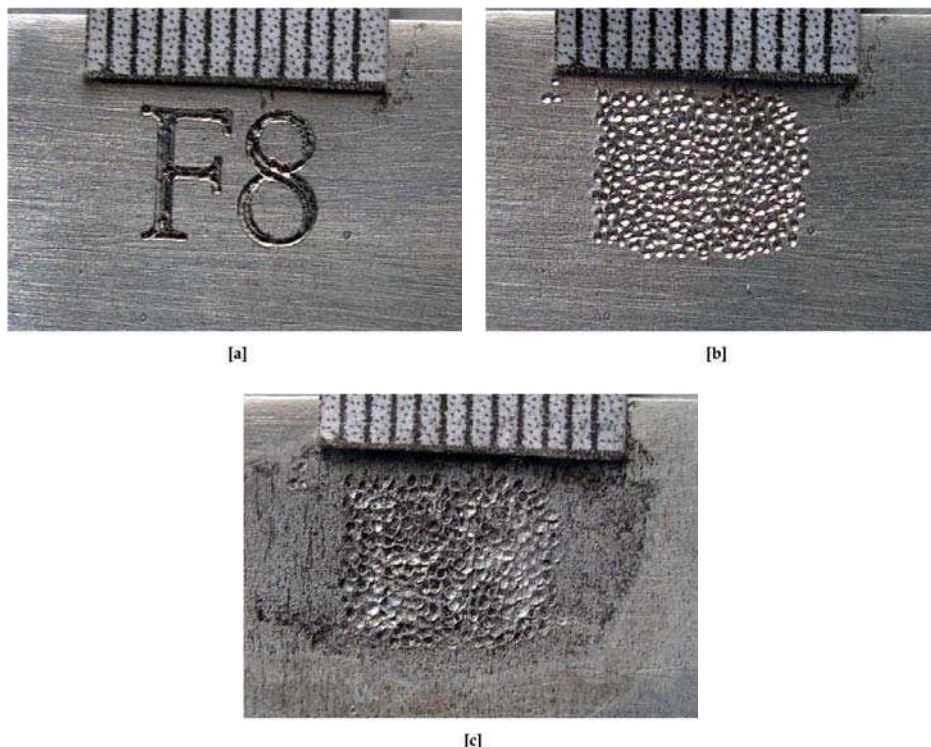
[a]

[b]

[c]

Fig. 9. a) The original engraved marks "F8" on Al-Zn-Mg-Cu alloy surface. b) The marks were obliterated by peening using centre punch. The obliterated marks "F8" were recovered distinctly with good contrast. Etching reagent: 10% aq. phosphoric acid by immersion. Etching time: 5 h.

The obliterated marks on *Al-Si alloy* surfaces responded effectively to the swabbing of 10% NaOH followed by 10% HNO_3. While NaOH etching restored the marks, the rinsing with HNO_3 removed the black deposit (Mondolfo, 1943; Petzow, 1976) caused by NaOH swabbing thereby increasing contrast. (For details see Uli *et al*, 2010). Interestingly, Hume-Rothery reagent recommended for etching Al-Si alloy surfaces in metallurgical literature (Mondolfo, 1943; Vander Voort, 1984) could not restore the obliterated marks.

Table 3 gives the reagents recommended for etching Al, Al-Zn-Mg-Cu and Al-Si alloys together with the application methods employed and also the characteristics of the recovered marks. The etchants described therein were sensitive enough to revealing the obliterated engraved marks where the deformation zones were shallow. Hence they could be applicable to recover other kinds of markings—*stamping, pin stamping and type wheel marking*—where the depths of serial numbers and the cold worked metal left behind would be considerable (Katterewe, 2006).

The experiments had also shown that alternate application of 60% HCl and 40% NaOH suitable for etching both Al (99% purity) and   Al-Zn-Mg-Cu alloy was not effective on Al-Si alloy. Further 10% aq. phosphoric acid produced good contrast on Al-Zn-Mg-Cu alloy alone. Significantly, 10% NaOH swabbing followed by 10% HNO_3 used for the etching of

Material	Etchant composition	Comments
Al (99% purity)	1. Hydrochloric acid 60% 2. Sodium hydroxide 40%	The two reagents were applied alternately, 3 min for each solution until the erased marks were retrieved. Between the applications the surface was wiped with a cotton wool and cleaned with acetone. For the engraved plates erased only upto the depth of erasure (0.02 mm) the obliterated marks appeared after about 7 min. For deeper obliterations the time of recovery increased upto 60 min. The procedure gave good contrast and sensitivity of the marks.
Al-Zn-Mg-Cu alloy	1. Hydrochloric acid 60% 2. Sodium hydroxide 40%	The two reagents were applied alternately, first with HCl for 3 min followed by NaOH for a similar period till the numbers became visible. Between the applications the surface was wiped with a cotton wool and cleaned with acetone. For engraved plates erased only upto the depth of erasure (0.02 mm) the number started to appear in about 2 min after application of 60% HCl. NaOH swabbing was followed that darkened the surface and further swabbing by HCl removed all the dark deposition showing the numbers clearly. When erasures were deeply produced the time of recovery increased upto 20 min. The procedure gave good contrast and sensitivity of the marks.
	10% aq. phosphoric acid	The obliterated area was surrounded by modelling clay. 0.5 ml of the reagent was filled inside the clay. The reagent was renewed every one hour. The dark discoloration was gently removed periodically using cotton applicator. The marks showed as faint outlines after a few minutes. After an hour or more the marks appeared white in colour. Immersion time: 1-16 h, dependent on the depth of erasure of the marks. The time needed to recover marks was varying and not consistent when the experiments were repeated. Constant monitoring was required, as immersion for longer time destroyed the marks. Recovered marks were good in contrast and sensitivity.
Al-Si alloy	1. 10% Sodium hydroxide, 2. 10% Nitric acid	The two solutions were applied alternately by swabbing the obliterated surface, first, with the NaOH for 3 min and then HNO$_3$ for 1 min. The surface was wiped with a cotton wool and cleaned with acetone in between the applications. For engraved plates erased only upto the depth of engraving (0.02 mm) faint marks started to show after 3rd min of the reagent application and clearer marks appeared during the 25th min of application. For deeper obliterations the time of recovery increased upto 40 min. The procedure produced marks with good contrast and sensitivity.

Table 3. Reagents for etching Al (99% purity), Al-Zn-Mg-Cu and Al-Si alloys for restoration of obliterated marks. The reagents were sensitive enough to recover the marks on the engraved plates erased down 0.02 to 0.04 mm below the engraving depth. The marks produced by the reagents were also reproducible.

Al-Si alloy did not produce desirable results for Al (99% purity) and Al-Zn-Mg-Cu alloy. It was thus significant that some etchants which worked rather well for some Al alloys had no effect on others. Hence in forensic restorations an evaluation of the composition of the alloy should be made before selecting the reagent.

The researchers have also demonstrated that the depths of recovery of obliterated marks decreased when a new number was overengraved after erasing the original number. For example in the case of Al (99% purity) surfaces the recoverable depth was 0.04 mm when the numbers were erased by grinding. However, when the marks were erased by grinding and a new number was engraved in their place, the recoverable depth decreased to 0.02 mm (Izhar *et al*, 2008). Similar effects were observed with the other two Al alloys (Bong & Kuppuswamy, 2010; Uli *et al*, 2010) and also with steel surfaces (Azlan *et al*, 2007). The results only indicated that the deformation zone from the original engraving was not affected by the new overengraving up to certain levels, but beyond this depth the original deformation was lost. Though not definite, it appears that some overlapping of the new zone with the original deformation did occur at a certain depth. The practical significance of this observation in real cases is that the sensitivity of the etching technique is lowered where a new number is overstamped after removing the original marks.

The marks obliterated by centre punching were recoverable only when the depths of punches were not deep enough. During our experiments many centre punched marks could not be recovered. The centre punching produced a series of holes which were closely spaced on the original marks. It was found that when the holes were within the limits of depths of original engraving the recoveries were successful.

5. Problems for future research

There have been constant efforts to identify effective etchants to reveal marks on aluminium surfaces which are finding increasing engineering applications. The problem encountered is one of poor contrast between the recovered numbers and background metal surface. The work on engraved marks has identified some sensitive and contrast producing reagents for etching Al (99% purity), Al-Zn-Mg-Cu and Al-Si alloys. However, the reagents restored those marks that were erased only upto a few micrometres depth. Further when new deformation was introduced over the original engraving, the contrast of the marks became poorer. Hence further refinements are to be considered in order to optimize the sensitivity and efficacy of the current methods. In recent years different aluminium alloys are being introduced in the firearms and motor vehicle industry. There is a need to carry out experiments to identify appropriate etchants for these surfaces. It is also desirable to conduct independent researches on restoration of stamping, pin stamping, engraving and type wheel marks in order to assess the recoverable erasure depths, as no work has been directed in this direction so far. Similar works however are available for steel surfaces (Turley, 1987; Wightman & Matthew, 2008).

Modern pistols and motor vehicles use laser engraved serial numbers and barcodes. Laser engraving of valuable jewellery may also be encountered in practice. These laser etched marks are difficult to be restored until the present time, as the process removes the metal by vaporization from intense heat produced by the laser during engraving. There will be no plastic deformation zone caused by such effect as in the case of stamping or engraving a number. The heat affected zone produced has different hardness than the rest of the metal. A more recent study (Klees, 2009) to determine the obliterated laser engraved markings on

Metallographic Etching of Aluminium and Its Alloys for Restoration
of Obliterated Marks in Forensic Science Practice and Investigations
349

ferrous frames of firearms using scanning electron microscopy and x-ray mapping proved quite ineffective. When the heat affected zone was removed, only some ghost remnants were restored in backscattered imaging. The method could not provide surface analysis to contrast the roughness differences produced during laser etching. Relief polishing, as noted earlier was also not effective for deep obliterations. Hence newer methods to exploit the hardness differences between heat affected and unaffected area should be addressed in the near future.

It is known that if a new serial number is directly stamped onto the smoothed/erased area of the old one, the recovery is impossible because of overlap of the two (old and new) deformation zones (Polk & Giessen, 1989). The present author experienced during his experimental work that when fresh engraved marks were made over the original ones some characters accidentally overlapped; such overlapped *original* characters could not be recovered. However, when the newer overengraved marks were slightly displaced from the previous number, recovery was full/partial dependent on the degree of overlap [Figs. 2. b), 5. b) & 8. b)]. It is worthwhile that this problem is addressed by material scientists for a good solution that will have far reaching implications in serial number restoration in forensic science practice.

6. Conclusions

This chapter has presented information on serial number restoration in forensic science investigations and also etching and other techniques pertaining to the visualization of obliterated marks on aluminium surfaces. Appropriate etchants from the many that are recommended for aluminium surfaces were identified. Some were sensitive enough to develop obliterated engraved marks, where the deformation zones were minimal of the order of tens of micrometers. They presented the marks with good contrast. Their etching responses on different aluminium surfaces are illustrated. Etching techniques are simple to employ and extremely useful in serial number restoration. They have revealed plastic deformation zones with great sensitivity and reproducibility.

Development of more sensitive etching methods for different kinds of aluminium alloys used in modern automotive components and firearms should be undertaken in the future. Also work remains to be done to find suitable techniques to raise obliterated laser etched marks. Further quantitative information is needed to establish the relationship between the depths of engraved/pin stamped/laser engraved marks and the respective depths upto which these marks can be restored.

Restoration of obliterated marks by chemical etching is both an art and a science. High quality surface preparation, selection of appropriate etchants, dexterity, alertness and care exercised during etching of the specimen, good observational skills to interpret the faintly recovered marks and mastery over closeup/macrophotographic techniques are all fundamental to the successful restoration of obliterated marks in forensic science practice and investigations.

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Metallographic Etching of Aluminium and Its Alloys for Restoration
of Obliterated Marks in Forensic Science Practice and Investigations

351

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Aluminium Alloys, Theory and Applications

Edited by Prof. Tibor Kvackaj

The present book enhances in detail the scope and objective of various developmental activities of the aluminium alloys. A lot of research on aluminium alloys has been performed. Currently, the research efforts are connected to the relatively new methods and processes. We hope that people new to the aluminium alloys investigation will find this book to be of assistance for the industry and university fields enabling them to keep up-to-date with the latest developments in aluminium alloys research.

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