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Recovering Erased and Obliterated Marks on High Strength Aluminum Alloy Surfaces by Etching Technique

Dissertation submitted in partial fulfillment for the Degree of Bachelor of Science in Forensic Science

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CERTIFICATE

This is to certify that the dissertation entitled

Recovering Erased and Obliterated Marks on High Strength Aluminum Alloy Surfaces by Etching Technique

is the bonafide record of research work done by

Miss Bong Yeu Uei

during the period December 2008 to April 2009

under my supervision.

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TABLE OF CONTENTS

Contents **Page**

LIST OF TABLES, CHARTS AND FIGURES

ABSTRACT

The current study describes the results of the experiments conducted on restoration of erased and obliterated marks on high strength aluminum alloy surfaces. The use of this alloy is on increase in the field of vehicle and firearm manufacturing. Serial numbers imprinted on these items are removed by criminals for several reasons. Recovering these erased or obliterated numbers is necessary in the solution of criminal cases. Hence, a series of experiments was designed to identify the sensitivity and efficacy of some of the metallographic reagents which could be used to recover marks on these surfaces.

The work consisted of preparing several high strength aluminum alloy plates and engraving marks on them using a computer controlled engraving machine "Gravograph". The marks were then erased to different levels down to the depth of the engraving. Five etching reagents were applied on the erased plates by either swabbing or immersion method to test their sensitivity and reproducibility. The results have shown that reagents i) 60 % HCl and 40 % NaOH and ii) 10 % phosphoric acid were able to produce good results, which were also reproducible.

Experiments were also performed by erasing the engraved marks until the marks were totally removed and later the erased area was over-engraved with some new marks. Also, experiments of obliteration of the original marks by centre-punching were conducted. In both cases of over-engraving and obliteration the above two reagents were successful to bring back the original numbers.

INTRODUCTION

Serial numbers are markings made on valuable items such as vehicles, firearms and jewellery for identification purpose. These serial numbers can be used to distinguish items of similar appearances (Petterd, 2000). In forensic science, serial number restoration is very important in the investigation of stolen vehicle cases and cases involving firearms (Nickolls, 1956; Heard, 1997). In some instances recovery of such original marks provides an important investigative aid.

Removal and obliteration of serial numbers are conducted by criminals in order to hide the true identity of the valuable items. The reasons for the removal include (Petterd, 2000):

- The perpetration of fraud, such as portraying an item to be of a different value when making an insurance claim
- To hide the origin of items used in the commission of a crime
- To prevent positive identification of stolen property

Vehicle forgery and stolen vehicles are one of the most serious crimes occurring throughout the world. In Malaysia, cases of stolen vehicles are very common as shown in Table 1:

Year	2005	2006	2007
Total cases	66,927	82,287	85,058

(http://www.rmp.gov.my/)

Table 1: Stolen Vehicle Cases in Malaysia

From Table 1, it is clear that there is an increasing trend in the number of vehicle theft cases from year 2005 to year 2007. Before these vehicles are sold, the criminal will remove or obliterate the identification numbers by various methods. These methods include filing or grinding, peening, over-stamping, centre-punching, drilling, welding, sanding etc (Heard, 1997; Stauffer & Bonfanti, 2006). Hence, restoration of the removed or obliterated serial number on the vehicle is necessary in order to solve the case. In addition, crimes which involve firearms especially murder cases also can be solved, if the obliterated serial numbers of the guns are restored successfully. From the serial numbers, the police will be able to detect the owner of the firearms or vehicles.

Several methods, either destructive or non-destructive methods can be used to restore these numbers, depending on the type of metals used. Among several recovery methods, chemical etching is the most sensitive technique.

The purpose of this study is to find out the most sensitive chemical reagents for restoration of erased numbers on high strength aluminum alloy surfaces. This aluminum alloy becomes more and more common in manufacturing of chassis of vehicles, because it is lighter compared to other metals such as steel. Some examples of cars made by aluminum alloys are Audi A8 (Audi, 2009) and Honda NSX (Wikipedia, 2009). In addition, many firearms like Ruger P85 pistol (Brown, 2001), Czechoslovakian P-01 pistol (Gunpedia, 2009), S&W Special AirWeight and AirLite revolver (Smith and Wesson, 2009) use aluminum alloy frames. Ergal, a hard aluminum alloy (AA 7075) also is used for manufacturing firearms (Bond, 2008).

There is not much published work for restoration technique for aluminum surfaces. In future, it is believed that more firearms and vehicles will be manufactured using aluminum alloys, hence identification of the most effective and sensitive etching reagent becomes necessary.

Theory behind Serial Number Restoration

Under the observation of microscope, metals are polycrystalline in structure. They have consisted of irregularly shaped crystals, or grains, which form when molten metal cools to the point of solidification. Between these crystals are interlocking regions, known as grain boundaries (Heard, 1997).

Whenever a stress is applied onto the metal, these crystals will be deformed, resulting in two different zones namely elastic deformation zone and plastic deformation zone. Elastic deformation is non-permanent, whereas plastic deformation is permanent. The depth of metal affected by the compressed crystalline structure depends on the type of metal and the force applied (Pork and Giessen, 1975; Stauffer and Bonfanti, 2006).

Restoration of serial numbers is only possible when the material onto which they were present still contains a plastic deformation due to their original presence. If obliteration is done until the number has just been removed, the plastic deformation zone which is the compressed region containing the altered crystalline structures will still be present and restoration is still possible (Stauffer and Bonfanti, 2006).

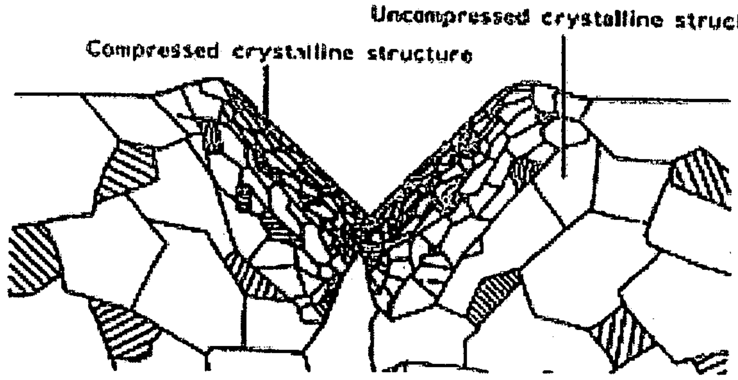

Figure 1: Cross section view of deformation of crystalline structure of metal (Heard, 1997)

Deformation of crystal structure alters its hardness, strength, magnetic, electrical and chemical properties. Therefore, the rate of reaction between etching chemicals and the metal can differ in the damaged and undamaged areas. A visible contrast resulted from differential reflection or scattering of light from the damaged area compared with that from the undamaged area are created (Heard, 1997).

Types of Markings and Serial Numbering

Serial numbers can be applied in a variety of ways, depending on the surface to be marked. Methods applied to metals and plastics surface may be different. The major methods used to mark metal surfaces are given as follows (Nickolls, 1956; Katterwe, 2006; Petterd, 2000; Stauffer and Bonfanti, 2006):

1. Die Stamping

   This technique can be done by using machine or by hand (hammer punches). An 'inverse positive' of the character is created, before it is stamped onto the metal surface under sudden pressure. Indented character is left behind. The depth of the impression relies on the pressure applied and the properties of the metal.

2. Stylus/pin marking

   Stylus is used to make individual dots onto the metal surface by applying some pressure. These dots arrange into the pattern of the character. The difference between die stamping and pin stamping is that the former method only requires single stroke, while the latter technique requires multiple strokes.

3. Roll marking

   This method is very similar to die stamping but the applied pressure is slower and steadier. It is commonly applied when the metal could be damaged by sudden impact used in die stamping.

4. Type wheel marking

   Preprogrammed hydraulic press or computer-controlled type wheels are used for markings.

5. Engraving

   The metal is marked by using a tiny spinning head which cuts away the substrate, leaving marks which form the serial numbers. The machine which controls this process is known as Gravograph.

6. Scribe marking

   It is a combination technique of stylus marking and engraving, producing only microchips and controlled by computer.

7. Laser beam marking

   Computer-controlled intense laser beams scan through the metal surface and mark it. The amount of heat produced by the laser alters the structure of metal and vaporizes metal on contact. The marking process is very fast and the heat affected zone will depend on the intensity of the laser and scan speed.

8. Embossing

   This technique is similar to die stamping or rolling, but is used on thin metal plates. Raised print appearance is produced in this method where the die is pushed onto the plate from behind.

Previously, die stamping was commonly used in serial numbering. Presently other markings especially pin stamping, engraving and laser etching are widely used on different kinds of objects. The plastic deformation of stamped marks is more than engraving. In case of laser beam, there is no plastic deformation zone but heat-affected zone (HAZ). Hence more sensitive methods need to be developed for engraving and laser markings.

Serial Number Restoration Techniques

The methods of restoration include destructive and non-destructive techniques. Here listed are some common techniques used in forensic laboratory (Polk and Giessen, 1975; Heard, 1997; Petterd, 2000; Stauffer and Bonfanti, 2006).

A. <u>Destructive techniques</u>

1. Chemical etching

   This is a very common and sensitive technique used in most crime laboratory nowadays. Many researches have been done on this method for different metal surfaces. Chemical reagents are applied by swabbing them onto the metal surface.

2. Electrolytic etching

   This is a modification of chemical etching which involves addition of electrical current in etching process. The metal to be etched is used as anode while wet cotton cathode is used to swab onto the metal surface.

3. Heat treatment

   Heat is applied onto the metal surface until the metal glows in a light cherry red color. The advantage of heat treatment is the process is faster than chemical etching. The temperature of heating varies and depends on the type of metal and depth of impression. Kehl (1949) suggested that chemical etching technique is applied after heat treatment.

4. Ultrasonic Cavitation

   Metals are placed into a water bath which is excited by an ultrasonic sound frequency but the method is only suitable for small objects. The numbers are formed by formation of bubbles.

B. Non-destructive techniques

1. Magnetic particle method

   Specimen is magnetized and then sprayed with fine magnetic particles which outline the obliterated number, if restoration is successful.

2. Relief polishing

   Restored number appears as a relief when the sample is polished, if there is a very different hardness between the bottom of erased marking and the rest of material.

REVIEW OF LITERATURE

Many studies had been conducted on the recovery of erased numbers on metal surfaces. These studies were carried out in order to understand the principle and theory behind recovery and also different methods used to restore serial numbers on different types of metal.

Kehl (Kehl, 1949) discussed in greater detail the etching reagents, etching mechanisms and etching procedures in his classic book "Metallographic Laboratory Practice". He explained that for a metal to be satisfactorily etched, the reagents to be used should take full cognizance of the composition of the specimen of interest. Hence, the selection of etching reagents must be done with careful consideration. Whenever an etching reagent is applied to a metal, the structural details are revealed because differently oriented grains have different rates of solution in the etching reagent. Regarding the etching procedure, he explained that different etching reagents required different methods by which the specimen was to be etched (immersion, swabbing etc.). In addition, the time of etching and the temperature of etching also depended upon the etching reagent and the type of metal surfaces.

Nickolls (Nickolls, 1956) described three types of markings which included cast marks, engraved marks and punched marks. He mentioned that underlying metal of engraved marks was not seriously disturbed compared to punched or stamped marks. In order to restore the erased marks, he suggested Fry's reagent for steel surface, Villella's solution for aluminum alloy and Hume-Rothery's reagent for high-silicon aluminum alloys. He also explained the depth of distorted or altered material was very small and does not exceed 1 mm. The ghost fragments should be recorded once it was recovered since it might appear quickly and then disappear during the restoration process.

Chisum (Chisum, 1963) noted that the standard etching reagents for iron and other metals reacted too vigorously with aluminum, producing blurred results. He suggested 1N NaOH, and 0.1M $HgCl_2$ in 0.1N HCl for restoration of serial numbers on aluminum and aluminum alloy and the working time average was about 20 minutes.

Polk and Giessen (Polk and Giessen, 1975) described metallurgical background of serial number recovery. The atoms within metals, known as grains, are densely packed together to fill space. The metal first becomes elastically strained and would return to its original shape when force is applied to it. However, as the force increases, the metal's elastic limit is exceeded, causing permanent deformation. Generally, when a number is stamped onto the surface of metal, the number consisted of a visible indentation, a plastically deformed region surrounding and defining the indentation, and an elastically strained region bordering the plastic region. The plastic region has deformed by dislocation motion and the metal near to the die has been pushed to the side and above the original surface. This region contained residual elastic stresses which were constrained from relaxing back to its original condition by opposing forces in the adjacent plastic region which had undergone a permanent change of shape. The amount of plastic flow and the depth to which the plastic region extends below the indentation depend upon the shape of the die and the depth of the indentation. In addition, they also described chemical or electrochemical serial number recovery methods on metal surfaces.

Voort (Voort, 1984) discussed the criteria for a good etching reagent. These included producing good results, simple in composition, inexpensive, easy to prepare, stable during use or storage, safe to be used and should not produce noxious odor. Besides, he also suggested 10 % phosphoric acid for restoration of aluminum alloys in addition to 20 mL HCl, 20 mL HNO_3, 5 mL HF, 40 mL water and 15 mL ethanol.

Heard (Heard, 1997) discussed different methods for removal of serial numbers, theory behind number restoration, etching procedure and etching reagents for different types of metal. The removal methods explained by him included filing or grinding, peening, over-stamping, centre punching, drilling and welding. Regarding the chemical reagents, he also suggested Villella's reagent and Hume Rothery's reagents as suggested by Nickolls (Nickolls, 1956) for aluminum alloy. Besides chemical etching methods, Heard also discussed magnetic particle method, electrolytic method and ultrasonic cavitation for number restoration.

Collins (Collins, 1999) discussed modern markings and serial numbering methods which included conventional stamping, pin stamping, laser etching and electro-chemical marking. In conventional stamping, the serial number is marked onto flat metal surface. In contrast to conventional stamping, pin stamping allows marking of surfaces that exhibit an irregular contour. The impact pin which is made by tungsten carbide and driven pneumatically or electromagnetically is used to mark serial numbers. The depth of deformation is lesser in pin stamping marks. In laser etching marking method, the characters are so shallow that they are visible to the eye only and hardly to feel by finger. The depth of deformation for laser etching will depend on the intensity of the laser and scan speed. On the other hand, electrochemical marking did not affect the metal below the actual depth of the mark and had no effect on the metal part that was being marked. Once the marking process was complete, the work piece had suffered no stress or change to its composition.

Petterd (Petterd, 2000) in his study discussed the reasons of removal of serial numbers, serial number marking methods, removal methods and restoration methods. In addition to serial number marking methods described by Collins (Collins, 1999), Petterd also explained about rolling, engraving and embossing methods. In terms of removal method, the author listed out filing, grinding, sanding and peening. There are two major methods employed for recovering obliterated serial numbers on metal surfaces which are 'chemical etching' and 'heat treatment'. Any visible characters should be noted immediately, as it is quite common for different characters to appear at different times during the restoration. It is also common for some characters to disappear shortly after becoming visible. The significant advantage of heat treatment is the speed, in which it is faster than chemical etching method. In heat treatment, heat is applied directly to the obliterated area until the metal glows a light cherry red. This results in the release of the residual tensile stresses, and allows, the compressed area to bulge above the surroundings. Heat treatment has been used with limited success on aluminum alloy substrates. Besides, the author also discussed magnetic particle method, X-rays, and ultrasonic cavitations. He suggested 10% NaOH, Hume-Rothery's solution, 25% HNO_3, and alternating 10% NaOH and 10 % HNO_3 on aluminum alloys.

Brown (Brown, 2001) restored serial number restoration on Ruger P series aluminum alloy frames, using acidic ferric chloride chemical etching reagent. The reagent was added onto the metal surface using saturated cotton tipped swab. The reagent was then allowed to effervesce for 1 to 2 minutes. The reagent was removed by spraying with distilled water. The numbers appeared after several cycles of application of this chemical reagent. The numbers were seen as darker 'ghostly' lettering. Holding the frame against a light background and tilting the frame to an oblique angle to the observer seemed to help increase the contrast between the darker lettering and the lighter metal of the frame.

Crowe and Morgan-Smith (Crowe and Morgan-Smith, 2005) reported the restoration of jeweler's mark in gold jewelry using *aqua regia* chemical solution. *Aqua regia* is made by mixing one part of nitric acid and three parts of hydrochloride acid. The etching solution was applied onto the gold surface by swabbing technique. The numbers were successfully restored after few cycles of swabbing.

Katterwe (Katterwe, 2006) also discussed various marking methods, techniques of obliteration of serial numbers, principle of deformation and restoration, and restoration methods. In addition to the marking methods that Collins (Collins, 1999) and Siegel (Siegel, 2000) discussed, he explained about roll marking and scribe marking. Techniques of obliteration noted by Katterwe were the same as that found in the previous literatures. For chemical etching technique, the chemical used in restoration method depended on the composition of the metal. They ranged from simple alkaline solutions to more complicated mixtures, which result in an oxidation or reduction reaction with the metal. The author noted down 10% Sodium hydroxide as the most common etching solution for aluminum alloys. The result of chemical etching can be photographed using oblique lighting which is able to produce best images. It is also necessary to test the best angle for the incident light.

In a recent work by M. Azlan et. al. (M. Azlan et. al., 2006), a specific metallographic reagent was found to be effective in restoration of erased engraved marks on steel surfaces. Research was conducted on restoration of obliterated engraved marks on low carbon (0.1 % C) steel surfaces. The metal was mechanically engraved with numbers using Gravograph. The numbers were later erased to different depths using sand paper. Chemical etching methods were used to restore these erased numbers. Eight etching reagents were tested using swabbing technique. They found that the reagent containing 5 g copper sulphate, 60 mL water, 30 mL concentrated ammonium hydroxide and 60 mL concentrated hydrochloride acid was the best reagent for restoration of erased engraved mark on low carbon steel surfaces. This etching solution revealed the numbers to a depth of 0.04mm below depth of engraving.

Silva and Santos (Silva and Santos, 2008) used relief polishing method in recovering obliterated laser engraved serial numbers in firearms. The firearms used in the experiment were aluminum alloy framed. At first, the surface was manually grinded or polished using abrasive paper numbers 600, 1200 and water. The scratches on the surface were removed at a faster rate than the marks produced by the laser due to difference of hardness in the regions where the number was engraved and the remaining region of frame. Any change at the region of interest was observed using stereomicroscope and the image was captured. However, for the frames which were seriously damaged or with scratches so deep, the serial numbers failed to be restored.

Izhar et. al. (Izhar et. al., 2008) conducted experiments on recovering obliterated engraved marks on pure aluminum surfaces by etching technique. Among eight reagents tested, 60% hydrochloride acid and 40 % sodium hydroxide were found to be the best reagents. These reagents were most sensitive to the aluminum surfaces, produced excellent contrast between the restored number and the background, and are able to restore the erased numbers up to 0.04 mm below the depth of engraving. The restored marks were permanent and were distinctly clear even after 2 years. On the other hand, using the same reagents, marks were restored when the numbers were erased 0.03mm below the depth of engraving and over-engraved with new numbers. In addition, the authors also found that thickness of metal plates affected the restoration depth. By increasing thickness from 0.61 mm to 1.5 mm, the recovery depth increased from 0.04 mm to 0.06 mm. They also found that deformation zone from the original engraving did not get affected by the new over-engraving up to certain levels, but beyond this depth the original deformation was lost.

Wightman and Matthew (Wightman and Matthew, 2008) investigated the relationship between the force applied and the depth at which restoration could be achieved after erasure of the die-stamped mark on steel surfaces. In this study, the authors used Fry's reagent with a composition of 90 g of copper chloride, 120 mL of concentrated hydrochloride acid and 100 mL of water. The results had shown that greater was the initial penetration, greater the depth at which the stamp mark could be recovered. However, doubling the stamp depth would not double the distance at which the mark could be recovered. They also conducted experiments on obliteration of stamped marks using chisel and the result was reproducible after removing 1.6 mm of metal (0.6mm of original imprint and further 1 mm). Regarding aging of etching solution, they found Fry's reagent became less reactive after long storage and the time required to restore the marks increased as the time of storage increased.

In another paper "Development of etching paste", the same authors developed etching paste using alumina powder. Fry's reagent was mixed with alumina powder and the paste was covered on the steel specimen. The paste is thixotropic, that is the paste would flow whilst it was agitated with the rod, and then ceased to flow when agitation ceased. Therefore, it was easy to ensure the paste covered the test area but did not overflow. At suitable time intervals the paste was wiped off, observation made and more paste added. Immersion techniques also were conducted to compare the difference between immersion and etching paste. The result had shown that etching paste was able to give better results. This etching paste was very suitable for the uses of serial numbers restoration when dealing with larger items such as vehicles.

Siaw and Kuppuswamy (2009) investigated the most sensitive chemical etching reagents for medium carbon (0.31% C) steel surfaces on chassis metal of a Mazda car. The experimental procedure was similar to that followed by Azlan et. al. and Izhar et. al. Seven etching reagents, mostly copper containing compounds were tested on the surface using swabbing techniques. The results had shown that Fry's reagent comprising 90g cupric chloride, 120 mL hydrochloride acid and 100 mL water was the most sensitive reagent for this metal. By comparing this result with that of M. Azlan et. al. (M. Azlan et. al., 2006), it was very clear that carbon content in steel should be considered when choosing the right chemical etching reagents.

From the above published work, it can be concluded that restoration of marks are possible when the marks are erased up to certain levels, where the deformation area is still present. Different chemical reagents and different techniques should be used to restore the erased markings depending on the metal surfaces. Further, suitable lighting and attentiveness are needed to view the restored marks. It is also established that the composition of etching reagent is specific to the composition of the metal.