Forensic Science International 208 (2011) 66–73

Contents lists available at ScienceDirect

Forensic Science International

journal homepage: www.elsevier.com/locate/forsciint

A survey of some metallographic etching reagents for restoration of obliterated engraved marks on aluminium–silicon alloy surfaces

Norjaidi Uli, R. Kuppuswamy *, Mohd. Firdaus Che Amran

Forensic Science Programme, School of Health Sciences, Universiti Sains Malaysia, Kampus Kesihatan, 16150 Kubang Kerian, Kelantan, Malaysia

ARTICLE INFO

Article history:
Received 9 July 2010
Received in revised form 17 October 2010
Accepted 2 November 2010
Available online 9 December 2010

Keywords:
Forensic science
Serial number restoration
Macroetchants
Chemical etching
Plastic deformation in metals
Aluminium–silicon alloy
Aluminium engine blocks

ABSTRACT

A brief survey to assess the sensitivity and efficacy of some common etching reagents for revealing obliterated engraved marks on Al–Si alloy surfaces is presented. Experimental observations have recommended use of alternate swabbing of 10% NaOH and 10% HNO_3 on the obliterated surfaces for obtaining the desired results. The NaOH etchant responsible for bringing back the original marks resulted in the deposition of some dark coating that has masked the recovered marks. The coating had been well removed by dissolving it in HNO_3 containing 10–20% acid. However, the above etching procedure was not effective on aluminium (99% purity) and Al–Zn–Mg–Cu alloy surfaces. Also the two reagents (i) immersion in 10% aq. phosphoric acid and (ii) alternate swabbing of 60% HCl and 40% NaOH suggested earlier for high strength Al–Zn–Mg–Cu alloys [23] were quite ineffective on Al–Si alloys. Thus different aluminium alloys needed different etching treatments for successfully restoring the obliterated marks.

Al–Si alloys used in casting find wide applications especially in the manufacture of engine blocks of motor vehicles. Hence, the results presented in this paper are of much relevance in serial number restoration problems involving this alloy.

1. Introduction

Metallographic etching techniques are extensively applied in forensic science laboratories to recover obliterated serial numbers on the chassis and engine of a stolen motor vehicle, or a firearm involved in a crime [1–5]. These identifying marks are removed from the metal components by mechanical means such as grinding or filing in order to prevent their identity. Visualization of the erased numbers provides important forensic evidence during criminal investigations. Over the past many years a large number of techniques using chemical etchants were developed empirically for many metals and alloys [6–16]. The mechanical inhomogeneities in a metal or an alloy that were introduced during stamping/engraving a number are revealed during etching because they react at inherently different rates with the reagents. This results in the manifestation of the obliterated number. Chemical etching of specimens is a straightforward, simple procedure that is easily mastered. Results with etchants are usually predictable and reproducible [14].

Etching techniques for obliterated stamped marks for iron and steel have been documented more thoroughly than any other metal because of the greater use of these in automotive engine and chassis and also firearms. There has been frequent tampering of serial numbers on these items. However, in recent years, forensic examiners encounter cases involving aluminium alloys in motor vehicle parts and more especially in the frames of firearms. Automobile parts have been manufactured with Al alloys instead of steel components to reduce the weight of the car. Modern firearms use high strength aluminium alloys for their frames [17,18]. More recently pin stamping, engraving and laser etching are applied for serial numbering on articles [4,19]. Compared to steel aluminium is a difficult metal to be etched for serial number restoration.

There have been a few publications in the past dealing with the recovery on aluminium surfaces [17,18,20,21]. The techniques described therein had their own limitations.

More recently Izhar et al. [22], and Bong and Kuppuswamy [23], have reported successful restoration of obliterated engraved marks respectively on pure aluminium and high strength Al–Zn–Mg–Cu alloy surfaces. They found that alternate swabbing of 60% HCl and 40% NaOH presented itself to be the desirable procedure for both the above aluminium surfaces. Bong and Kuppuswamy [23] further noted that for etching Al–Zn–Mg–Cu alloy immersion in 10% aqueous phosphoric acid for long hours produced better contrast than alternate swabbing of 60% HCl and 40% NaOH. The current paper is an extension of these works and reports the efficacy and

* Corresponding author. Tel.: +60 9 7677583; fax: +60 9 7677515.
E-mail address: rkuppuswamy@kb.usm.my (R. Kuppuswamy).

0379-0738/$ – see front matter © 2010 Elsevier Ireland Ltd. All rights reserved.
doi:10.1016/j.forsciint.2010.11.005

Table 1
Chemical composition of the alloys in % weight.

Brand of the engine block	Copper	Magnesium	Silicon	Iron	Manganese	Zinc	Lead	Nickel
Yamaha	2.54	0.13	10.60	1.08	0.24	0.65	0.085	0.17
Honda	2.18	0.20	10.76	0.84	0.18	0.75	0.054	0.076

sensitivity of some common reagents to etching with special reference to aluminium–silicon alloys. Al–Si alloys are used in castings and they find wide applications in the manufacture of engine blocks of automobiles.

2. Experimental

2.1. Alloy sample

Two old motorcycle engine blocks made of aluminium alloys – one from Yamaha and the other from Honda – were purchased for this work. Their chemical composition (% weight) is given in Table 1.

Of the two engine blocks mentioned above etching experiments were carried out first on the Yamaha engine block. Later the results were tested on the alloy block of Honda engine which is of similar chemical composition.

The Al alloy blocks of both Yamaha and Honda engines were cut into several small plates for the etching experiments.

2.2. Engraving the plates and their obliterations

The procedures employed in this section were similar to those reported elsewhere [22,23]. However, they are briefly described here.

2.2.1. Engraving the alloy surfaces

The Yamaha aluminium–silicon alloy plates were engraved with a combination of alphanumerical characters “M8” using a computer-controlled machine “Gravograph”. The depths of the engraved marks were found to be approximately 0.02 mm (20 μm). The engraved marks were obliterated in three ways: (i) erasure by grinding, (ii) peening using centre punch until the marks were not discernible and (iii) erasure by grinding followed by overengraving some new marks.

2.2.2. Obliteration of the marks by grinding

A series of five plates of five different erasure depths was prepared for etching. The first engraved plate was erased by grinding just until the visible engraving had been totally removed. The remaining four plates were abraded respectively to 0.01, 0.02, 0.03 and 0.04 mm below the bottom of the engraving depth. The plates were erased with P80, P150 and finished with fine grade P320 sand papers. Since nine reagents were to be tested, nine series of the above plates totalling 45 were prepared for the experiments.

2.2.3. Obliteration of the marks by centre punching

The marks “M8” were obliterated by peening using centre punch until the marks were no longer decipherable to the naked eyes.

2.2.4. Obliteration of the marks by overengraving

The original marks “M8” were removed by grinding to a depth of 0.02 mm below the bottom of engraving using abrasive papers. Some new marks, “W6” were overengraved in the erased area.

All the obliterated plates were appropriately labelled.

2.3. Etching reagents

Nine reagents recommended by earlier researchers and available in open literature for etching aluminium and its alloys were tested on those plates obliterated by grinding (refer Section 2.2.2). The list of reagents, their composition and the sources of references are given in Table 2.

2.4. Restoration technique

No polishing of the alloy surface was done, as the plates were sufficiently smoothened during grinding of the marks by sand papers [refer Section 2.2.2]. The plates were cleaned using acetone to remove debris, dust and other particles, if any. Following this the surfaces were etched by either swabbing or immersion method. Swabbing was done by dipping a cotton bud in the reagent and swabbing it onto the obliterated surface. The surface was swabbed gently, evenly and with constant force. This was to ensure that the erased engraved marks were fully restored.

In the immersion method the obliterated surfaces were immersed in the reagent for several hours. It was applied to the phosphoric acid reagent alone.

For recovering the marks obliterated by centre punching and overengraving, the most effective reagent identified for recovering erased marks by grinding was applied following the procedures of restoration.

3. Results

3.1. Restoration of marks erased by grinding

Table 3 provides the results of the etching experiments. As can be seen therein the etching reagents from numbers 5–9 did not produce either good contrast or any effect on the erased area and were found to be quite ineffective in the restoration procedures. While etching reagent 4 (acidified ferric chloride) produced some ghost marks, reagent 3 (1 N NaOH and 0.1 M HgCl in 0.1 M HCl) faintly recovered the marks erased until the depth of engraving. The etching reagent 2 (10 g NaOH in 90 ml H_2O) was quite successful to recover the marks erased up to 0.03 mm below the engraving depth and it was very sensitive. However, the marks appeared as long as the reagent remained on the surface of the plate. Once the reagent dried up and cleaned up by acetone the marks disappeared. These effects are seen in Fig. 1. The reagent 1 (alternate swabbing of 10% NaOH and 10% HNO_3) [11] was sensitive and also recovered the marks with revealing contrast. The marks recovered by this reagent are seen in Fig. 2.

Table 2
Etchants used on aluminium–silicon alloy surfaces for recovering the marks erased by grinding.

Etching reagent	Reagent composition[a]	References
1	1. 10% sodium hydroxide 2. 10% nitric acid	Petterd [11]
2	1. Sodium hydroxide 10 g 2. Water 90 ml	Katterwe [4] Petterd [11] Kehl [13] Vander Voort [14] Petzow [15]
3	1. 1 N sodium hydroxide (1 mol/l)	Polk and Giessen [6]
	2. 0.1 M mercuric chloride aq. (0. 1 mol/l) in 0.1 N hydrochloric acid (0.1 mol/l)	Chisum [20]
4 Acidified ferric chloride	1. Ferric chloride 25 g 2. Hydrochloric acid 25 ml 3. Water 100 ml	De Forest et al. [3] Brown [17]
5	10% aq. phosphoric acid	Vander Voort [14] Bong and Kuppuswamy [23]
6 Nital	1. 1 Part nitric acid 2. 9 Parts of ethanol	Cunliffe and Piazza [2]
7	1. 60% hydrochloric acid 2. 40% sodium hydroxide	Peeler et al. [21] Izhar et al. [22]
8	1. Nitric acid 25 ml 2. Water 75 ml	Petterd [11] Petzow [15]
9	1. Cupric chloride 15 g in 100 ml of water	Vander Voort [14]
(Hume–Rothery etch)	2. 50% HNO_3	Mondolfo [16]

[a] While making the reagents always add acid to water while stirring. Never store concentrations of nital (ethanolic solutions in nitric acid – reagent number 6 shown in this table) greater than 5%, as at 10% it is comparable to rocket fuel in its volatility.