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# THE EFFECT OF DOUBLE SHOT PEENING AND NITRIDING ON THE DIE SOLDERING BEHAVIOUR OF H13 AND Cr-Mo-V TOOL STEEL

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## ABSTRACT

In this study, H13 tool steel and Cr-Mo-V steel were treated by two different types of surface treatments, i.e. double shot peening with nitriding and single shot peening. Samples were dipped into the molten aluminum alloy ADC12 as a simulation of the die casting process and held there for 0.5, 5, and 30 minutes. Several characteristics were analyzed, including surface hardness, microstructure observation, and identification of elements on the intermetallic layer formed. The results of the research showed that H13 steel treated by double shot peening with nitriding had higher surface hardness (1402 VHN) than when treated by shot peening only (536 VHN). A similar tendency emerged with the Cr-Mo-V steel, which had 1402 VHN and 503 VHN after treatment with double shot peening with nitriding and the single shot peening process. In addition, with a dipping time of 30 minutes, the H13 steel treated by double shot peening with nitriding produced a lower average thickness of the compact intermetallic layer. Moreover, double shot peening did not form a broken intermetallic layer, while single shot peening formed one (91.66 µm). Likewise, the Cr-Mo-V steel treated by double shot peening with nitriding produced a thinner compact intermetallic layer than single shot peening, 22.2 µm vs. 27.77 µm, as well as a lower average thickness of the broken intermetallic layer, 40.2 µm vs. 113 µm. This indicates that material treated by double shot peening with nitriding could minimize the occurrence of die soldering.

*Keywords:* Die casting; Die soldering; Double shot peening; H13 steel; Nitriding

## 1. INTRODUCTION

High pressure die casting is widely used for the formation of aluminum in the automotive industry, and can produce between 20,000 and 250,000 products per month (Shankar & Apelian, 2002). However, in practice, the die material comes out sticking to the casting products. This phenomenon is called die soldering, and can damage or require replacement of the mold. It occurs due to the reaction between the die material and the molten Al alloys, which forms an intermetallic compound Fe-Al-Si on the die material surface. This reaction causes material to stick to the die and makes it difficult to open. Such damage can decrease or even stop the production process because of the defective products produced (Han & Viswanathan, 2003).

Erosion of the die surface during early stages of die soldering is closely related to the nature of the die material, especially the hardness of the mold material (Shankar & Apelian, 1999; Shankar, 2000). Some surface treatment processes that can improve the hardness of the surface

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are shot peening and nitriding. Shot peening can produce residual stress and nitriding will increase the hardness of the surface of the material (Hassani et al., 2014). Therefore, in order to determine the effect of these surface treatment processes on die soldering, in the present study a double shot peening surface treatment was applied before and after the nitriding process.

Shot peening prior to nitriding will provide the appropriate nitrogen gas diffusion into the surface during the nitriding process, thus resulting in a more homogeneous hardness distribution. However, shot peening after nitriding is expected to eliminate the brittle compound layer, eliminate micro cracks, and increase micro hardness on the surface of the material, as the shot peening treatment produce cold worked effect with high surface hardness, density and compressive stress (ASM Handbook, 1994). Thus the surface treatment engineering is expected to minimize erosion on the walls of the die which would otherwise occur in the early stages of die soldering.

## 2. EXPERIMENTAL PROCEDURES

### 2.1. Materials and Surface Treatment Processes

The materials used in this study are modified H13 steel and Cr-Mo-V steel with the chemical compositions shown in Table 1. The two materials are tool steels with different alloy compositions, and are commonly used as die casting mold materials.

Table 1 H13 and Cr-Mo-V steel chemical composition (weight %)

| Composition (Wt %) | C | Mn | Si | Cr | Mo | V |
|---|---|---|---|---|---|---|
| H13 Steel | 0.39 | 0.4 | 1.00 | 5.30 | 1.40 | 0.9 |
| Cr-Mo-V Steel | 0.35 | 0.5 | 0.20 | 5.00 | 2.30 | 0.5 |

Prior to the nitriding process, the steels were treated with a hardening process to increase their hardness to 45-48 HRC. Parameters such as preheating, hardening, quenching, and tempering temperatures follow the materials' standards (ASM Intl., 1991).

At the beginning of the hardening process, both steel materials were preheated twice for 30 minutes at temperatures of 650$^{o}$C and 850°C in order to avoid thermal shock and cracking during austenizing. The austenizing temperature reached 1025$^{o}$ C for 60 minutes, followed by air cooling. This was followed by tempering for 2 hours at temperatures of 580$^{o}$ C, 590$^{o}$ C, and 600$^{o}$C, respectively, to achieve a hardness of 45-47 HRC.

There were two path variations of the surface treatments done to the materials:

1. *As hardened* → *shot blasting* → *shot peening* → finish (SB-SP).
2. *As hardened* → *shot blasting* → *shot peening* → nitriding → *shot peening* → *tempering* → finish (SP-N-SP).

The shot blasting process was performed to clean the surface so that impurities such as oxides were removed by fine particles of SiC. Shot peening was conducted after the shot blasting process to create higher surface tension, by firing off the H13 and Cr-Mo-V steel samples with a fine steel ball (55-58 HRC hardness and 0.3 mm diameter) with a pressure of 460 kPa for 20 minutes.

After the shot peening, gas nitriding was conducted in a vacuum furnace, so the nitrogen would diffuse into the steel surfaces, using ammonia and nitrogen gas (50% $NH_3$, 50% $N_2$) at a temperature of 520$^{o}$ C for 10 hours, followed by tempering at a temperature of 540$^{o}$C for 120 minutes. The second shot peening was conducted after the nitriding process was completed, and followed by Rockwell hardness testing in accordance with ASTM E-18.

Prior to the dipping test, steel samples were prepared in two sizes, 20×10×10mm and 30×10×10mm, for microstructure observation and immersion tests. To observe the occurrence of the die soldering effect, the samples were dipped into a molten aluminum alloy, and the sample weight loss was measured before and after the dipping process. The samples with higher weight loss after the dipping process were analyzed as having a higher tendency to have die soldering effects during the die casting process.

### 2.2. Melting and Dipping Test in Molten Aluminum Alloy

ADC12 aluminum melting was carried out at a temperature of 680$^{o}$C. Table 2 shows the alloy composition.

Table 2 ADC 12 molten aluminium composition

| Aluminum Alloy Composition (Weight %) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Al | Si | Cu | Mg | Fe | Mn | Ni | Zn | Pb | Sn | Ti | Cr |
| Bal | 10-12 | 2.5-3.5 | 0.3 | 1.3 | 0.5 | 0.5 | 1 | - | 0.3 | - | - |

Dipping tests were done for durations of 30 seconds, 5 minutes, and 30 minutes, to simulate the die casting process. A weight loss test was conducted, to determine the effect of shot peening and nitriding on the reaction and the formation of intermetallic phases as a result of the diffusion and loosing of Fe to the aluminum. Samples were characterized using the micro Vickers hardness and macro Rockwell hardness of the surface, layer thickness measurements, and their microstructure was measured with an optical and Field Emission Scanning Electron Microscope (FE SEM) to observe the morphology and topography of the die soldering layers. The surface chemical composition was measured with an Electron Dispersive Spectrometer (EDS) to analyze the composition of the element at the layers and the intermetallic compound developed at the surface.

## 3. RESULTS

### 3.1. Effects of Double Shot Peening and Nitriding on Surface Hardness

A Vickers hardness testing method was conducted to determine the effectiveness of the surface treatment with a nitriding process as well the shot blasting and shot peening, for both the H13 and Cr-Mo-V steel samples. Figure 1 shows the relationship between hardness and distance from the surface of the H13 and Cr-Mo-V steel. The tendency of hardness decreases with increasing distance from the surface of the material. From the hardness testing on H13 and Cr-Mo-V steel between variables SB-SP and variable SP-N-SP, it was found that the hardness on the surface of the SB-SP variables only reached 536 VHN for H13 steel and 503 VHN for Cr-Mo-V steel, with no significant differences between surface hardness and the interior. Hardness in the variable SP-N- SP reached 1402 VHN on the diffusion layer near the surface of both types of steel. The hardness of the material decreased from 1402 to 759 VHN with increasing distance from the surface, up to a depth of about 145.37 µm for H13 steel and 162.96 µm for Cr-Mo-V. This indicates that the average effectiveness diffusion layer for H13 steel and Cr-Mo-V steel formed around 145.37 µm and 162.96 µm, respectively.

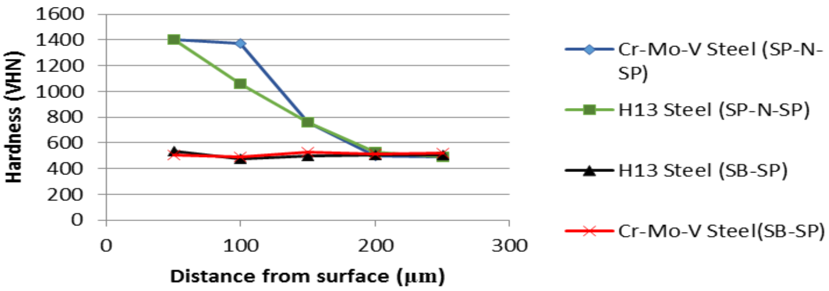


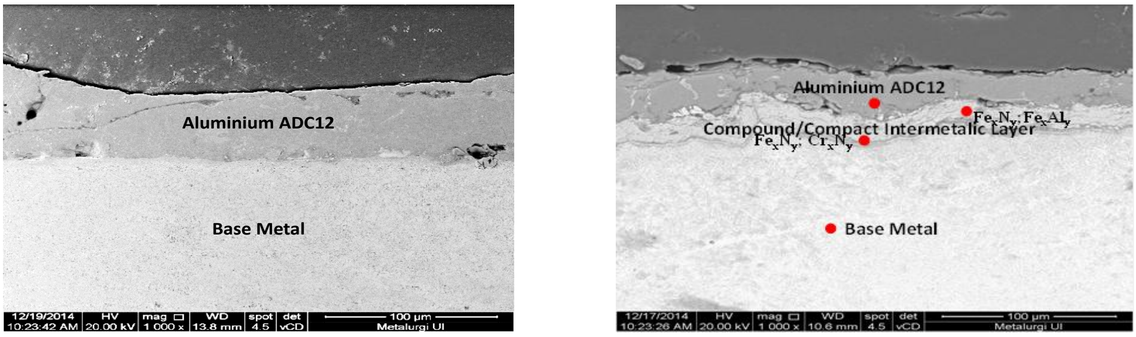
Figure 1 Hardness distribution of H13 and Cr-Mo-V steel after SB-SP and SP-N-SP surface treatments, and the relation of hardness to distance from the surface

The high hardness of the SP-N-SP process occurs because nitrogen diffuses into the material based on an interstitial basis, and reacts to form compounds with nitride former elements that are dispersed evenly as nitride (Slima, 2012). Because the process were diffusion process, the hardness value will decrease as the distance from the surface increases.

On the other hand, the hardness of the H13 and Cr-Mo-V steel with an SB–SP variable showed no difference with increased distance from the surface. The shot peening process causes a strain-induced martensitic formation, and thus only slightly increases the hardness on the surface (Hassani et al., 2014).

### 3.2. Effect of Double Shot Peening and Nitriding on Intermetallic Compound Formation

After a dipping test in molten ADC12, the intermetallic compound thickness and composition were observed by a Field Emission Scanning Electron Microscope (FE SEM) and an Electron Dispersive Spectrometer (EDS) to determine the die soldering effect.



(a) (b)

Figure 2 FE SEM photograph of the effect of contact time with liquid aluminum ADC12 on H13 Steel (SP-N-SP) for: (a) 30 seconds; and (b) 30 minutes

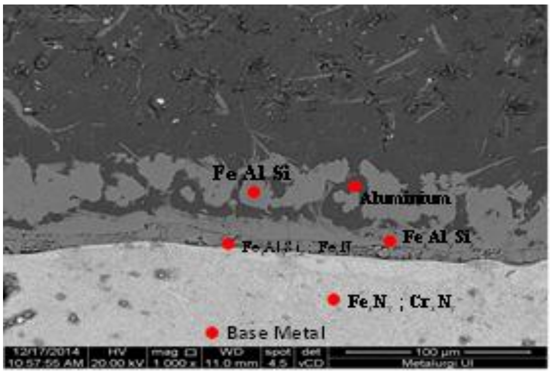


Figure 3 FE SEM photograph of the compound layer of SP-N-SP at a dipping time of 30 minutes for Cr-Mo-V Steel

Prediction of the compound layer formed on the steel surface is based on the phases available according to the Fe-Al phase diagram, and was analyzed by Electron Dispersive Spectrometer (EDS) testing. Figure 2 shows an optical microscope photograph and FE-SEM of the H13 SP-N-SP steel samples after dipping them into molten ADC12 for 30 seconds and 30 minutes at a temperature of 680$^{o}$ C. Figure 2a shows that a dipping time of 30 seconds does not form an intermetallic layer. EDS analysis indicated that it is an aluminum layer.

For the 30-minute dipping time, a thick intermetallic layer formed and is indicated as $Fe_2Al_5$, namely a compact intermetallic layer that is solid. Due to the long immersion time, Fe diffuses out and reacts with molten aluminum to form a compact intermetallic layer. The metal diffusion coefficient in the semisolid intermetallic phase achieve 3–4 times greater levels than a pass through a solid phase (Suharno et al., 2007). At a dipping time of 30 minutes a gap forms between the material and molten aluminum alloys, showing the formation of soldering in the form of erosion pit.

Figure 2b and Figure 3 show the differences in the formation of an intermetallic layer on the H13 and Cr-Mo-V steel surface under the SP-N-SP treatment at a dipping time of 30 minutes. EDS analysis (Table 3) shows that in H13 steel, the compact intermetallic layer are $Fe_2Al_5$, CrxNy, and FexNy and are solid on the surface of the steel. On the Cr-Mo-V steels the broken intermetallic layers formed are α- (Fe, Al, Si) and are semi-solid on the surface of the steel. This layer also contained a compound indicated as FexNy and CrxNy, suspected to originate from the diffusion layer. Both materials showed no die soldering phenomenon because they only developed one intermetallic layer formation, while the die soldering effect is characterized by the formation of two intermetallic layers, one solid and one broken.

As shown in Figure 4 and Table 3, the H13 steel with SB-SP treatment has no phase of CrxNy and FexNy. There is the ternary FeAlSi phase of the broken intermetallic and compact intermetallic layer. This morphology indicates that soldering reactions occur. Soldering occurs if two layers are formed, an intermetallic layer (solid) and a broken intermetallic layer (semi-solid). If only one of the intermetallic phases are formed, this indicates no die soldering formation (Tang, 2007). The formation of one phase is due to the low Al concentration on the steel surface. From EDS analysis (Table 3**)**, the Fe content will decrease with increasing distance from the material surface. Likewise, the composition of Al decreases with distance closer to the steel.

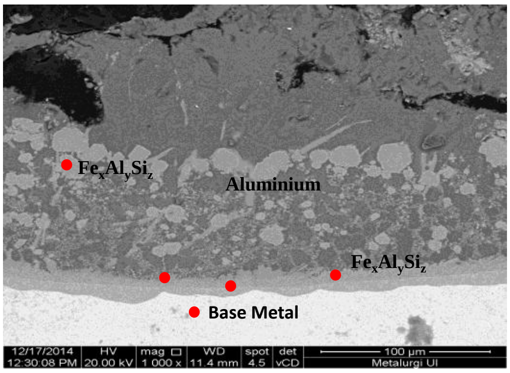
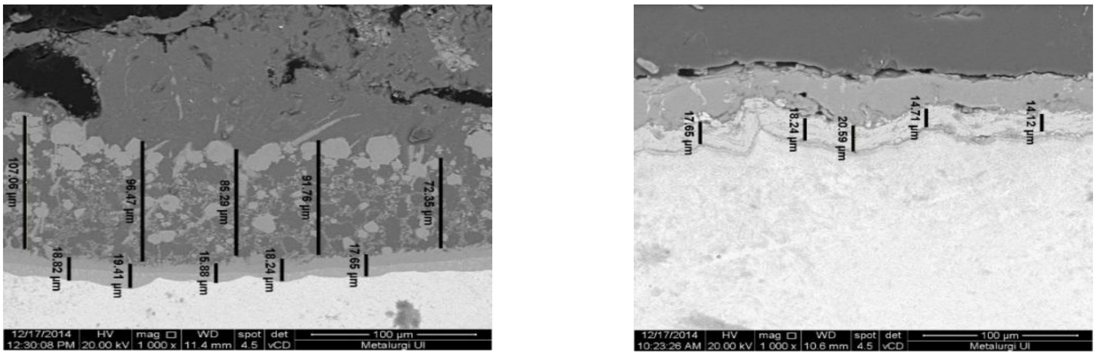


Figure 4 FE SEM photograph of H13 Steel with SB-SP surface treatment after being dipped for 30 minutes in a molten aluminum alloy

On the other hand, Figure 5b and Table 3 show that during SP-N-SP treatment, the binary phase formed between Fe with Al is FexAly, contained in the layer on the surface of the steel material commonly referred to as a compact intermetallic layer, is more solid and more resistant to soldering compared to the ternary phase α- (Fe, Al, Si) contained in the broken intermetallic layer in the SB-SP treatment.

Table 3 Phase predictions of the soldering layer developed during dipping tests in ADC12 liquid aluminum alloy

| Material | Dipping Time | Points | Composition (Weight %) | | | | | Compound Indication |
|---|---|---|---|---|---|---|---|---|
| | | | Al | Si | Fe | N | Cr | |
| Cr-Mo-V Steel (SB-SP) | 30 min | 1 | 82.14 | 14.33 | 2.83 | 0 | 0.7 | *α-(Fe,Al,Si)* |
| | | 2 | 68.37 | 23.66 | 6.89 | 0 | 1.08 | *α-(Fe,Al,Si)* |
| | | 3 | 71.83 | 11.5 | 0 | 16.43 | 0.23 | *Aluminium ADC12* |
| | | 4 | 52.84 | 24.13 | 0 | 23.03 | 0 | *Aluminium ADC12* |
| Cr-Mo-V Steel (SP-N-SP) | 30 min | 1 | 69.5 | 20.2 | 10.4 | 0 | 0 | *α-(Fe,Al,Si)* |
| | | 2 | 97.6 | 0 | 0.92 | 1.49 | 0 | *Aluminium ADC12* |
| | | 3 | 62.5 | 23.7 | 12 | 1.87 | 0 | *α-(Fe,Al,Si)* |
| | | 4 | 61.5 | 23.1 | 12 | 3.39 | 0 | *α-(Fe,Al,Si); FexNy* |
| | | 5 | 3.87 | 3.62 | 68.3 | 14.3 | 9.95 | *FexNy ; CrxNy* |
| | | 6 | 0.74 | 0 | 73.2 | 15.8 | 10.3 | *Base Metal* |
| H13 Steel (SB-SP) | 30 min | 1 | 67.9 | 17.15 | 12.6 | 0 | 2.35 | *α-(Fe,Al,Si)* |
| | | 2 | 98.37 | 0.56 | 0.68 | 0 | 0.39 | *Aluminium ADC12* |
| | | 3 | 82.1 | 14.68 | 2.58 | 0 | 0.64 | *α-(Fe,Al,Si)* |
| | | 4 | 62.78 | 18.64 | 17.5 | 0 | 1.09 | *α-(Fe,Al,Si)* |
| | | 5 | 1.84 | 3.82 | 83.27 | 4.25 | 6.82 | *αFe* |
| H13 Steel (SP-N-SP) | 30 min | 1 | 68.8 | 31.2 | 0 | 0 | 0 | *Aluminium ADC12* |
| | | 2 | 29.2 | 3.65 | 58.3 | 8.85 | 0 | *FexNy;*$Fe_2Al_5$ |
| | | 3 | 2.09 | 11 | 62.3 | 9.6 | 15 | *FexNy ; CrxNy* |
| | | 4 | 0.97 | 4.63 | 74.5 | 9.03 | 10.9 | *αFe* |
| | | 5 | 2.64 | 4.61 | 79 | 6.61 | 7.12 | *αFe* |



(a) (b)

Figure 5 Intermetallic compound thickness measurements by SEM, H13 Steel (SB-SP): (a) H13 (SP-N-SP); and (b) dipping 30 minutes in liquid ADC12

Intermetallic compound average thickness measurements by SEM for the H13 steel at a dipping time of 30 minutes are shown in Figure 5. The same method was applied for Cr-Mo-V steel. It was shown that H13 and Cr-Mo-V steel treated by double shot peening with nitriding produce a lower average thickness of the compact intermetallic layer, 17.1 µm and 22.2 µm, than in the single shot peening only process, 18 µm and 27.77 µm. Moreover, double shot peening with nitriding does not form a broken intermetallic layer, while single shot peening formed a broken intermetallic layer of 91.66 µm. Moreover, double shot peening with nitriding produced a lower average thickness of the broken intermetallic layer, 40.2 µm, than the single shot peening, 113 µm, for the Cr-Mo-V steel. This indicates that material treated by double shot peening with nitriding could minimize the occurrence of die soldering.

Phase prediction as the result of EDS analysis is shown in Table 3.

## 4. CONCLUSION

H13 and Cr-Mo-V steel exposed to a double shot peening treatment with nitriding produce a higher surface hardness, compared with single shot peening treatments. At a dipping time of 30 seconds, H13 steel does not form an intermetallic layer. Conversely, at 30 minutes dipping time, a thick intermetallic layer was formed. This shows that at 30 seconds dipping time, no soldering effects were indicated.

Die soldering occurrence is characterized by the formation of an intermetallic layer on the surface of the mold material. At the 30 minute dipping time, H13 steel and Cr-Mo-V Steel using a double shot peening treatment with nitriding (SP-N-SP) produced a compact intermetallic layer with lower average thickness, produced a thin or absent broken intermetallic layer, and had 60% less weight loss compared with single shot peening (SB-SP). This indicates that a double shot peening treatment with nitriding can minimize the occurrence of the die soldering effect.

## 5. ACKNOWLEDGMENT

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