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Die Soldering Phenomenon on the H13 Tool Steel with Shot Peening and Nitriding Surface Treatment

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Abstract. Die soldering occurs when molten aluminum sticks to the surface of a die material and remains there after the ejection of the part. This resulted in low productivity and economic value in the foundry industry. Nitriding surface treatment is considered as an effective way in enhancing the service life of AISI H13 steel dies and to prevent soldering effect. The focus of this paper is to investigate the influence of three different surface conditions in terms of roughness, gas nitriding and pretreatment prior to gas nitriding on the soldering effect. Three kind of samples made of AISI H13 steel were pretreated (quenched and tempered) and followed by : shot peened, gas nitrided and shot peening followed by gas nitriding, were immersed in liquid melted ADC 12 Aluminium alloy at 30 seconds, 30 minutes, 2 hours and 5 hours, at a constant temperature of 680^{o}C in a holding furnace. Characterizations on the surface of the steel were focused on the optical microstructure, microhardness profile, FE SEM observation and enegy dispersive spectrometry mapping. It was found that shot peening prior to nitriding gives a higher surface hardness and depth of nitride layer of H13 tool steel, 1140 HV (>70 HRC) and 120.5 µm, than the nitriding only process, 1033 HV (68 HRC) and 105 µm. The higher the hardness and depth of nitride layer expected would reduce the die soldering effect at the surface of the H13 tool steel dies. It was also found that the only shot peening treatment resulted in a tendency of soldering accompanied by the formation of intermetallic layers ; while soldering is not found on the nitrided and shot peened-nitrided samples.

Introduction

Quality of dies or molds in die casting process determines whether or not the quality of the resulting product. The biggest cost as much as 20% of the total production cost is the cost of die casting mold. **[1].** Therefore, the lifetime of the mold is a major concern. This relates to the efficiency of a die casting process. The more total products that can be produced before the mold has failed, it will be more economical die casting process. Die soldering is the most common failure and a major cause of downtime in the process of industrial Aluminum die casting [2].

Die soldering is a reaction between the surface of the mold with molten metal which cause the attachment of the molten metal into the mold surface. The phenomenon of die soldering will lead to mold damage, poor quality of cast products, and a decrease in productivity and efficiency of the die casting process. Therefore, if the formation of die soldering could be minimized, the lifetime of the mold and the efficiency of die casting process can be improved.

According to Shankar [2], the mold surface engineering is the most effective way to prevent the die soldering. The reason is because the die soldering is closely related to the dissolution of printed material on the surface of the mold [3]. Up to now,surface engineering method most widely done by the industries to reduce causes of diffusion soldering reaction is by nitriding [2]

The Nitride layer which formed in the nitriding process will prevent the direct contact between the mold with molten aluminum material so that the diffusion reaction between Fe and Al causes of die soldering can be minimized. Shot peening before nitriding expected to increase the thickness of the layer and the surface hardness after nitridisasi through an increase the rate of diffusion of nitrogen.

The most important stages in the process of die soldering is when the initial erosion of the mold surface by molten aluminum and the formation of intermetallic compounds. The mechanism of the formation of pitting can be caused by two things: first the existence of collision mechanically mold surface to molten aluminum, and both the presence of intergranular corrosion on the surface of the mold by molten aluminum [4,5]. Erosion or mechanical abrasion depends on the strength and hardness of mold material, while the intergranular corrosion depends on the chemical composition and microstructure on the surface of the mold [5, 6].

At the time of die soldering process, there are two opposing mechanisms, the first growth of the intermetallic layer at the interface due to reaction kinetics Fe / Al; and second dissolution of intermetallic layer on the molten metal cast which has a lower Fe content which causes the reduction of the thickness of the intermetallic layer. The relationship between dissolution with atomic diffusion of Fe and Al resulted in a decrease in weight (weight loss) and a decrease in volumetric (volumetric loss) of the steel mold [7].

Nitriding is a surface hardening process thermochemical material by forming a hard nitride alloy deposition on the surface, without the occurrence of phase changes during cooling to room temperature. In nitriding process, nitrogen atoms diffuse into the ferrite phase when the material is heated at a temperature 500-590oC (below the line AC1) and reacted with nitride-forming alloying elements on the surface of hard material forming nitride layer. Materials that can dinitridisasi is medium carbon steel (medium carbon steels) containing nitride-forming elements, such as Al, Cr, Mo, V, Ti, Nb, and Zr [8,9]

Shot peening is a cold working method on the surface of the material, which is done by continuously shots materia surfacel by spherical particles of small size and high speed so that produces a compressive residual stresses at the surface and cause indentation or indentations small rounded overlapping on the surface. Particles that are used can be either cast steel, cast iron, glass beads, or ceramic. It must be high precision controlled, like its roundness ball, shot intensity, shot particles, and also areas in the shot peening [10]. In this paper, the effect of nitrided samples with and without shot peening prior to nitriding to the die soldering effect is studied bye immersion test to liquid ADC 12 Aluminium Alloy . Sample with Shot peening only is also investigated for comparison purpose. All the samples were properly heat treated (quenched and tempered) prior to surface preparation and subjected to the same nitriding cycle.

Material and Experimental Procedure

The material used in this study is H13 tool steel measuring 85 x 75 x 10 mm which has been hardened to 45-47 HRC hardness have. The process consists of a hardened preheat temperature for 30 minutes at 650^{o}C and 850^{o}C, austenitizing at 1025^{o}C for 60 min, quenching with N2 gas pressure to 50-70^{o}C , and triple tempered for 2 hours at the temperature of 580^{o}C, 600^{o}C, and 580^{o}C. There are 3 variations of surface treatment condition were given the results of hardened tool steels, as follows:

a) *as-hardened* → *shot peening* (SP samples);
b) *as-hardened* → *shot blasting* → nitridisasi, → *shot blasting* → *tempering* (N samples);and
c) *as-hardened* → *shot blasting* → *shot peening* → nitridisasi, → *shot blasting* → *tempering* (SPN samples).a

Shot blasting uses particle green SiC (JIS 700), through a nozzle pressure of 476 kPa

Shot peening performed using spherical particles of steel with a hardness of HRC 45-48 and 0.3 mm diameter nozzle pressure 461 kPa. Shot peening performed for 20 minutes . Gas Nitriding performed using ammonia gas (50% NH3, 50% N2) at a temperature of 510oC for 5 hours, this is to produce a white layer thickness of less than 3 μm. Tempering performed for three hours at a temperature of 540oC.

Fig.1 Shot Blasting Machine (left) and Shot Peening (right)

Samples of surface treatment variations then cut to a size of 20x10x10 m for microstructure observation and hardness distributionsmeasurements. To analyze the differences in depth and hardness of diffusion layer between specimens treated by shot peening and without shot peening prior to nitriding , micro Vickers hardness testing was conducted. Immersion of samples into the ADC12 aluminum alloy is performed for 30 seconds , 30 minutes , 2 hours , and 5 hours at a constant temperature of 680oC in a holding furnace .To test the die soldering effects, observation and measurement of the presence of intermetallic phases by the sample weight loss before and after immersion . After immersing in liquid aluminium , intermetallic compounds at steel surface cleaned with NaOH , and then weighed again and compared with the weight before immersion .

Samples with high weight loss means high amount of iron have reacted with liquid Aluminium and more intermetallic compound in the steel surface. Tests were conducted in macro and micro hardness testing.The difference in the character of the surface microstructure was analyzed using optical microscope, FE-SEM. EDX are used to determine the elemental content of each nitride layer results.

Results and Discussion

H13 steel composition tested with Optical Emission Spectrometer, and the composition showed in table 1 belows

Table 1.Chemical Composition of Modified of H13 Steel (Weight %)

C	Si	S	P	Mn	Ni	Cr	Mo
0.406	1.00	0.03	0.055	0.351	0.113	4.830	1.35
Ti	Cu	Nb	V	Al			
0.003	0.086	<0.005	1.03	0.027			

Effect of Shot Peening to Maximum Hardness,the White Layer thickness, and effective depth of diffusion layer.

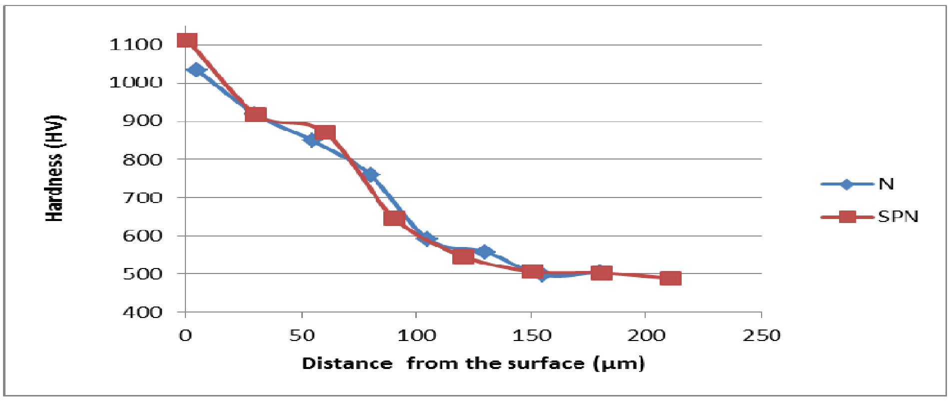
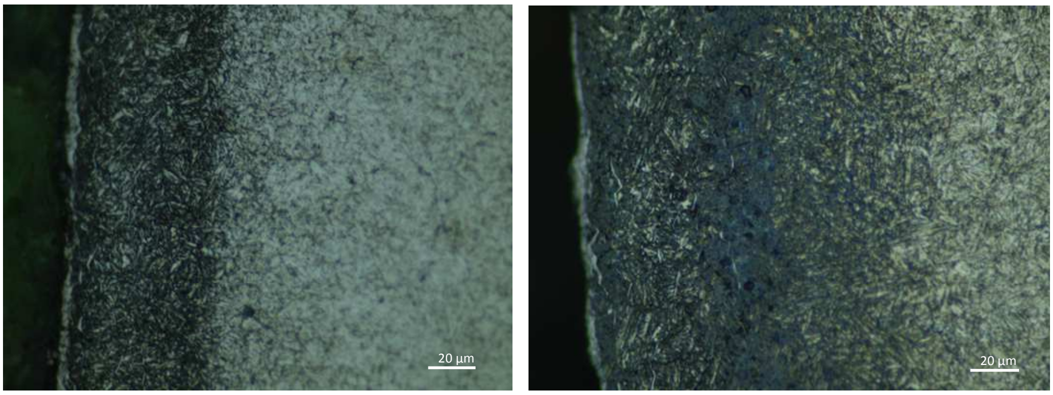


Fig. 2 Hardness Distribution of Nitrided Samples

Figure 2 shows the results of hardness testing nitride samples in the atmosphere of ammonia (50% NH3, 50% N2) for 5 hours at a temperature of 510oC. It Shows that both samples of N and SPN have the Nitriding hardening effect only to a certain distance from the surface and does not affect the hardness of the core or center. It was found that the average maximum hardness and the effective case depth of SPN samples have small higher value when compared with N samples. Maximum average hardness of N samples is 1033 HV (68 HRC) and the SPN samples is 1114 HV (> 70 HRC). While the effective depth of diffusion layer of samples N and SPN is 105 μm and 120.5 μm, as shown in figure 3. These results indicate that shot peening influence nitriding coating hardness.



(a) (b)

Fig.3 Optical Micrographs of nitrided samples without shot peening sample N (a), and microstructure of samples with shot peening prior nitriding, sample SPN (b) Etching Nital 3%

Figure 3 shows the diffusion layer of Shot Peened-Nitrided samples (SPN samples) which have higher thickness than Nitrided samples without Shot Peening (N samples).

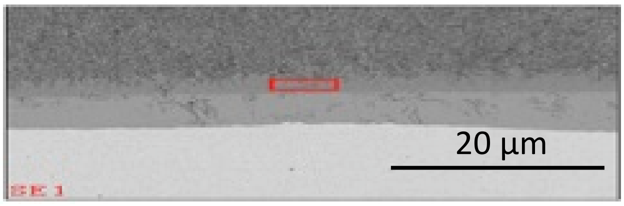
Effective depth of diffusion layer indicates higher diffusion rate of nitrogen . The rate of diffusion of an element is affected by two factors, temperature and activation energy of diffusion [11]. If the temperature and time of nitriding parameters are the same for two materials with different effective depth of diffusion layer, then it can be caused by differences in the surface diffusion activation energy of the material. This difference may be caused by two things. First, that the sample had shot peening will create more grain boundaries and dislocations that provide a shortcut for nitrogen atoms to diffuse more rapidly to the surface of the material [12-14]. As already known, the density of defects or dislocations and grain size are two important factors that enhance the diffusion process of nitrogen [11, 15, 16]. Second, the diffusion coefficient of nitrogen in bct-martensite phase is higher than in the fcc-austenite phase [12]. As mentioned earlier, the plastic deformation caused by shot peening process can cause strain-induced martensitic formation [17], the change of the metastable austenite to martensite due to plastic deformation. At the same time in nitriding, with more atoms of nitrogen that enters the surface of the sample which had been shot peened, means that diffusion rate of nitrogen increased and more nitrides developed at the surface.

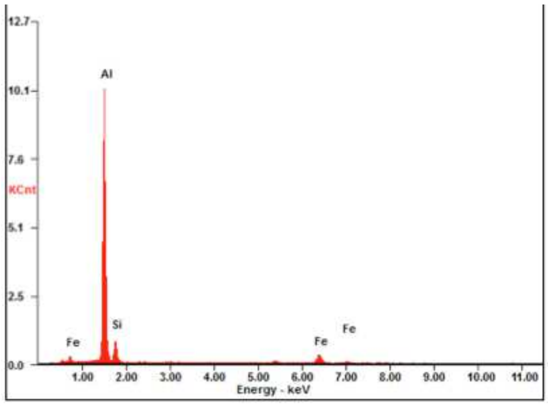
White layer is developed as the nitrogen phase ὲ (epsilon) which is formed due to the low nitrogen concentrations. With the higher diffusivity of nitrogen in SPN samples, which had been shot peened before nitriding process, the lower the precentage of nitrogen at the surface of the sample, the less phase ὲ developed and continue of to grow its thickness as nitrogen diffuses into the steel.The thickness of white layer of N and SPN samples were 6 μm and 3.1 μm.

Effect of Shot Peening on the formation of intermetallic compound and weight loss of AISI H13 steel at the immersion test.

The Immersion test results of AISI H13 steel in liquid aluminum alloy ADC12 , were shown at figure 4. The Phases of the samples after immersion test in the ADC 12 Aluminium Alloy observed by Scanning Electron Microscope (SEM) and were analiyzed by Phase prediction based on elements content as the result of Energy Dispersive Spectrometer(EDS) analysis .More Al at the intermetallic layer between steel and Aluminium Alloy predicted as FeAlSi Compound.

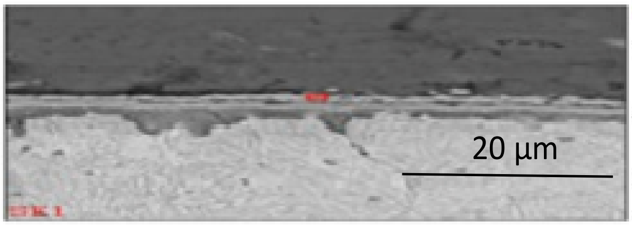
Element	Wt%	At%
AlK	71.23	75.50
SiK	19.30	19.65
FeK	09.48	04.85
Matrix	Correction	ZAF

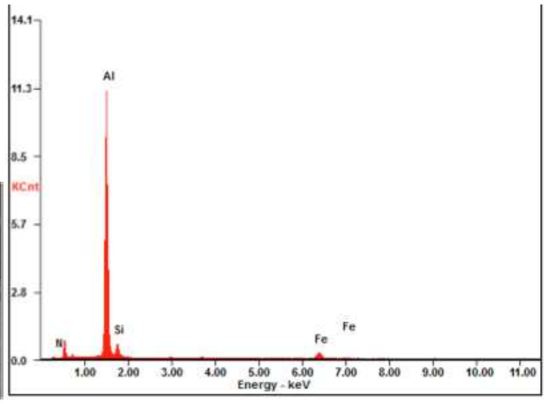




(a)

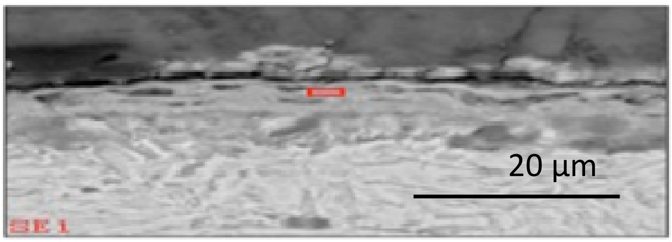
Element	Wt%	At%
NK	00.65	01.30
AlK	76.76	80.10
SiK	14.45	14.49
FeK	08.13	04.10
Matrix	Correction	ZAF

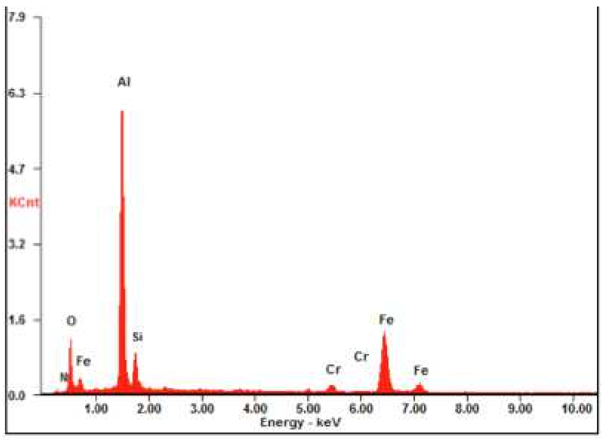




(b)

Element	Wt%	At%
NK	00.59	01.16
OK	17.29	29.87
AlK	44.25	45.34
SiK	09.84	09.68
CrK	02.03	01.08
FeK	26.01	12.88
Matrix	Correction	ZAF





(c)

Fig.4 SEM Micrographs and EDS analysis of Intermetallic layer on the surface of AISI H13 steel as immersion test results at 680 ^{o}C for 30 minutes , samples of SP (a) , N (b) , SPN (c)

Figure 4 shows Metallic compound at the AISI H13 steel surface with the different surface treatment after immersion test in liquid ADC 12 Aluminium Alloy. It showed that samples with shot peening prior to Nitriding surface treatment (SPN) have less FeAlSi compound layer as the compound layer has Aluminium content (44 wt%) compared to at SP and N samples were 71 and

76 wt%. Less compound FeAlSi layer at the steel surface correlated with more compact nitride layer and less Aluminium Alloy deposited and reacted with the steel surface, and will reduce the die soldering effects during die casting process. Weight loss measurement shows that SPN samples have less weight loss (0.09 gr) compared to weight loss of SP and N samples of 1.9 g and 1.42 gr. This also proven that Shot Peening prior to Nitriding surface treatment reduce the die soldering effect during Aluminium die casting process.

Conclusion

1. Nitriding of H13 tool steel in atmospheric environments of ammonia (50% NH3, 50% N2) for 5 hours at a temperature of 510^{o}C, showed an average maximum hardness condition of the surface by shot peening treatment preceded with nitriding, 1114 HV (> 70 HRC),greater than the surface conditions with nitriding only treatment , 1033 HV (68 HRC).
2. Immersion of H13 tool steel into Aluminum alloy ADC12 , Samples with shot peening prior to Nitriding surface treatment (SPN) have less FeAlSi compound layer as the compound layer has lower Aluminium content (44 wt%), compared to SP and N samples,71 and 76 wt%. It showed at SPN samples have only small number of intermetallic particles without soldering phenomenon. With increasing hardness of SPN samples before nitriding ,a large dislocation density due to the residual stress obtained from shot peening treatment on the surface, will reduce the phenomenon of die soldering to steel surfaces.
3. Microstructure of nitriding layer showed that the surface diffusion layer thickness are higher at samples with the shot peened before Nitriding , compared to the thickness of the steel surface which only have the Nitriding without shot peening.

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