

## A LOW TEMPERATURE ALUMINIZING TREATMENT OF HOT WORK TOOL STEEL<sup>1</sup>

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### Abstract

Conventional aluminizing processes by pack cementation are typically carried out at elevated temperatures. A low temperature powder aluminizing technology was applied to hot tool steel H13. The aluminizing treating temperature was from 550 to 620°C. Effects of temperature and time on the microstructure and phase evolution were investigated. Also, the intermetallic layer thickness was measured in the aluminized layer of a steel substrate. The cross-sectional microstructures, the aluminized layer thickness and the oxide layer were studied. Scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), glow discharge optical spectroscopy (GDOS) were applied to observe the cross-sections and the distribution of elements.

**Keywords:** Low temperature pack aluminizing; Aluminum oxide coatings; High pressure die casting; Reactive soldering.

### UM TRATAMENTO DE ALUMINIZAÇÃO À BAIXA TEMPERATURA PARA UM AÇO FERRAMENTA PARA TRATAMENTO A QUENTE<sup>1</sup>

### Resumo

Processos de aluminização por *pack cementation* são normalmente realizados em temperaturas elevadas. Usando uma tecnologia de aluminização por pó em baixa temperatura foi aplicada em aço ferramenta H13. A temperatura de tratamento de aluminização ficou entre 500 e 620°C. Os efeitos da temperatura e do tempo na microestrutura e na evolução das fases foi investigada segundo as medidas da espessura da camada intermetálica e a identificação das fases na camada aluminizada do substrato de aço. As microestruturas em secção transversal, a espessura da camada aluminizada e do óxido foram estudadas. Microscopia Eletrônica de Varredura (MEV), Espectrometria de raios-X por energia dispersiva (EDX), Espectroscopia óptica por descarga de brilho (GDOS) foram usados para determinar a distribuição dos elementos na secção transversal.

**Palavras-chave:** Aluminização em baixa temperatura; Revestimento de óxido de alumínio; Fundição em alta pressão; Soldagem reativa.

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## 1 INTRODUCTION

Die casting is a cost-efficient process to form cast products of aluminum, zinc, magnesium and copper based alloys.<sup>[1-3]</sup> The die life is a major consideration of the die casting process because a die may cost more than the die casting machine, depending on the complexity of the part being produced. Therefore, increasing the life of a die can reduce the cost of the parts being produced. The die failure modes, which limit the life and performance of die casting dies, can be classified as thermal fatigue cracking (or heat cracking), gross fracture, erosion and chemical attack including corrosion and soldering. Thermal fatigue cracking and die soldering are two major modes which lead to die failure in aluminum die casting.<sup>[4-7]</sup> Numerous past studies have focused mainly on the study of thermal fatigue cracking.<sup>[8-10]</sup> However, with the development of the die casting industry, increasingly more consideration is being given to die soldering, resulting in a notable increase in the efficiency and productivity of the die casting process. Soldering, or die sticking of aluminum, occurs during a die casting process when the molten aluminum „welds“ to the die surface, resulting in the damage of the die and in a poor surface quality of the casting. Two types of soldering are identified in the literature: the one that occurs at a high temperature due to chemical/metallurgical reactions between the molten aluminum alloy and the die and the other that occurs at a low temperature due to mechanical interaction. For the type of soldering occurring at high temperatures, it is generally acknowledged that soldering is closely related to the „washout“ of a protective film on the die surface. The washout occurs when the molten aluminum alloy enters the die and destroys the protective film (coating or lubricant) on the die. The molten aluminum then comes into contact with the die surface. The iron in the die substrate dissolves the melt whereas aluminum and other elements in the melt diffuse into the die. As a result, a layer of intermetallics is formed on the die surface. By applying a coating to the die surface to act as a physical barrier, reactive soldering can be prevented, thus reducing the machine down-time and the need for frequent polishing. A lot of researchers have begun to study the soldering phenomenon in their theoretical or experimental work and have reported significant improvements in the die performance with the application of surface treatments such as nitriding, thermo-reactive deposition (TRD), plasma-assisted chemical vapor deposition (PACVD), physical vapor deposition (PVD) and duplex treatments combining nitriding with physical vapor deposition (PVD) coatings.<sup>[11-15]</sup> The literature reveals that alumina layers could be formed on steels containing aluminum, such as Incoloy MA956, a ferritic oxide dispersion strengthened (ODS) Fe-Cr-Al alloy. Alumina ( $\text{Al}_2\text{O}_3$ ) has a better thermodynamic stability, with a larger amount of free energy of formation than iron oxides. Alumina-forming super alloys contain a sufficient amount of aluminum to form a protective alumina ( $\text{Al}_2\text{O}_3$ ) layer on the surface under high-temperature oxidation conditions.<sup>[16]</sup> In addition, researches on the application of oxidation on hot work tool steel (H13) are also reported in literature. Here, the formed layer of ferric oxide ( $\text{Fe}_3\text{O}_4$ ) would prevent the occurrence of reactive soldering on the tools used for aluminum die casting.<sup>[17]</sup> Oxidizing the Incoloy MA956 alloy has several advantages over oxidizing H13 steel. First, a single oxide layer, exhibiting better wear resistance, can be formed. Also, satisfactory results can be achieved by the oxidation process which uses oxygen from the air. Incoloy MA956 is more expensive than H13 tool steel, but Incoloy MA956 could replace H13 steel in the areas of the die that are subject to higher wear. The aluminizing process is normally applied at temperatures above 750°C. Thermal treatment at these temperatures applied for long periods of

time, typically 6–30 h, in a pack aluminizing cycle, can fatally degrade the mechanical properties of alloy steels as a result of microstructure changes associated with grain coarsening and carbide precipitation.<sup>[14,15]</sup> To prevent such degradation, the aluminizing process needs to be carried out at temperatures below 650°C. The process has the processing simplicity and flexibility required for treating components of various dimensions and geometries.

Therefore, this paper presents the results of a low temperature aluminizing treatment of H13 hot work tool steel. From the aluminum-iron phase diagram, we can determine the parameters of low temperature aluminizing which enables the introduction of aluminum into the substrate below the solubility of aluminum in iron without the formation of intermetallic compounds. This can be achieved only by low temperature aluminizing in the ferrite area and not by conventional high temperature aluminizing in the austenite area with the potential of aluminum below the limit of formation of intermetallic layers. Consequently, aluminizing was carried out at lower temperatures than the temperature of the previously carried out tempering of hot work steel (650°C). The aluminizing process took place in the mixture of aluminum powder, inert filler and various activators. Subsequently, oxidizing gas atmosphere containing either H<sub>2</sub>O or CO<sub>2</sub> was applied to oxidize the aluminum dissolved in the surface layer to obtain a compact layer of Al<sub>2</sub>O<sub>3</sub>, which is thermodynamically stable up to very high temperatures and which cannot be reduced by liquid aluminum.

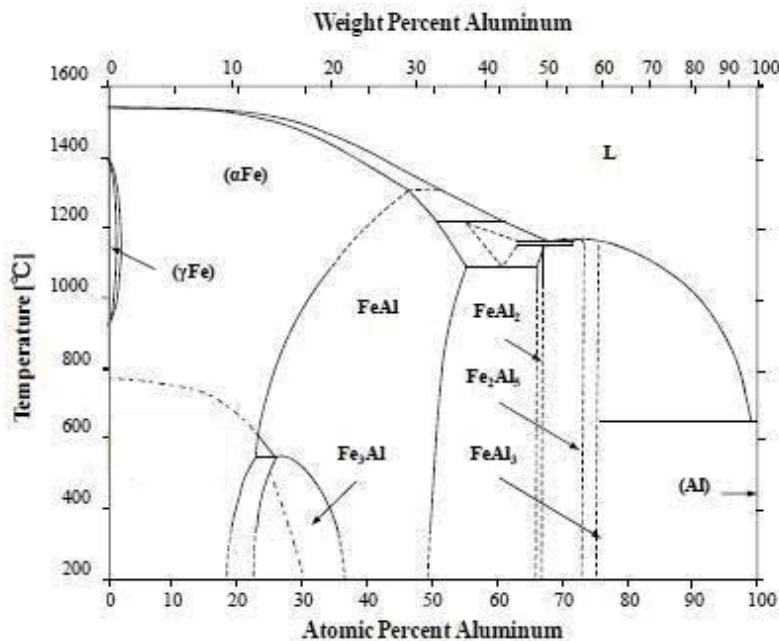


Figure 1. Aluminum-iron phase diagram.

## 2 EXPERIMENTAL PROCEDURES

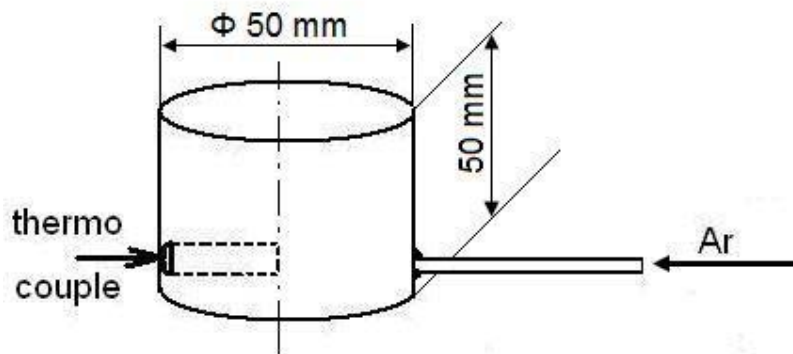
The substrate material used for investigation was a hot-work steel X37CrMoV5 1. The chemical composition of the material, analyzed by using bulk glow discharge optical spectroscopy, was as follows: 0.35 % C, 1.05 % Si, 5.0 % Cr, 1.3 % Mo, 0.32 %V. Specimens were prepared to dimensions of approximately 20 x 40 x 2 mm and then manually polished using SiC abrasive paper to a 600-grade and ultrasonically cleaned with acetone.

Aluminizing powder mixtures were prepared by weighing out and mixing appropriate amounts of aluminum powder - Al, filler Al<sub>2</sub>O<sub>3</sub> and halide salt as activator. The powders in the present investigation were supplied by SHERA, Germany (Al<sub>2</sub>O<sub>3</sub>) and KEMIKA, Croatia (aluminum powder- Al and halide activators). Three different halide salts, NH<sub>4</sub>Cl, AlCl<sub>3</sub> and NaCl, were assessed as possible activators for the intended pack aluminizing process at temperatures below 650°C. The pack composition for alumina coatings in all cases were as follows: 8 wt % of Al, 2 wt % of a halide activator and 90 wt % of Al<sub>2</sub>O<sub>3</sub>. The processing conditions are summarized in Table 1.

**Table 1.** Processing conditions of pack aluminizing

Samples	Activator	Temperature, °C	Duration, h
A	NH <sub>4</sub> Cl	620	4
B	AlCl <sub>3</sub>	550	4
C	NaCl	550	4

The substrate samples and pack material were placed in a special cementation retort in an argon atmosphere, Figure 2. They were then placed into an electric furnace, which was heated to 550°C and held at this temperature for 4 hours. The samples were then allowed to cool to room temperature.



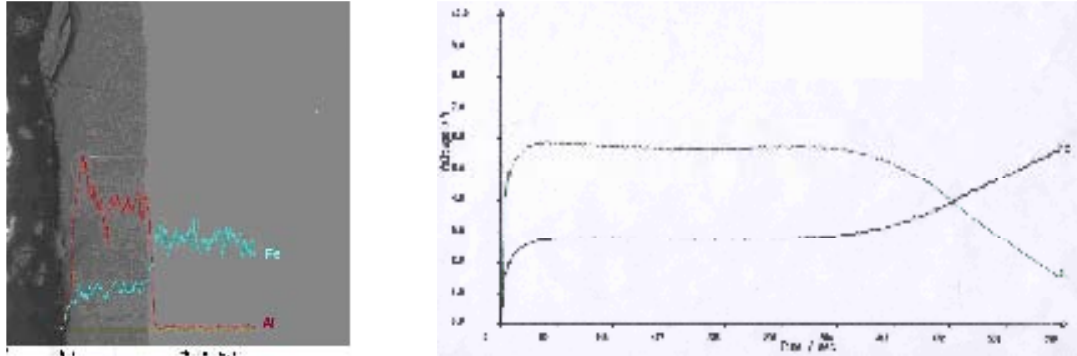
**Figure 2.** Schematic diagram of the special retort for powder aluminizing.

For metallographic examination, the specimens were cross-sections cut perpendicularly to the surface layer using a micro cutting machine and then mounted. To observe the microstructure of the surface coating layers formed by diffusion treatment, the cross-sections were mechanically polished using abrasive papers of grades 220 – 800, while the final polishing was carried out using diamond paste of up to 1 µm. The polished specimens were etched using 2 % NITAL at room temperature. The microstructure and chemical composition of cross sections of the coated specimens were also analyzed using scanning electron microscopy (SEM) TESCAN VEGA TC with energy dispersive spectroscopy (EDS) OXFORD INCA and glow discharge optical spectroscopy (GDOS) LECO SPECTRUMAT- 750.

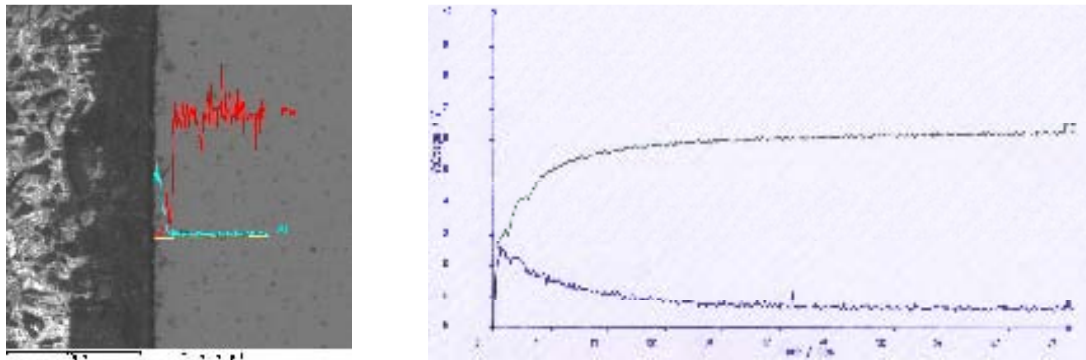
### 3 RESULTS AND DISCUSSION

With A samples, treated with the NH<sub>4</sub>Cl activator, aluminized at 620°C in the period of 4 hours, a layer of 50 µm in thickness was formed. From the Al and Fe distribution throughout the layer one can see that the content of elements is constant, which indicates the intermetallic connection between iron and aluminum, Figure 3.

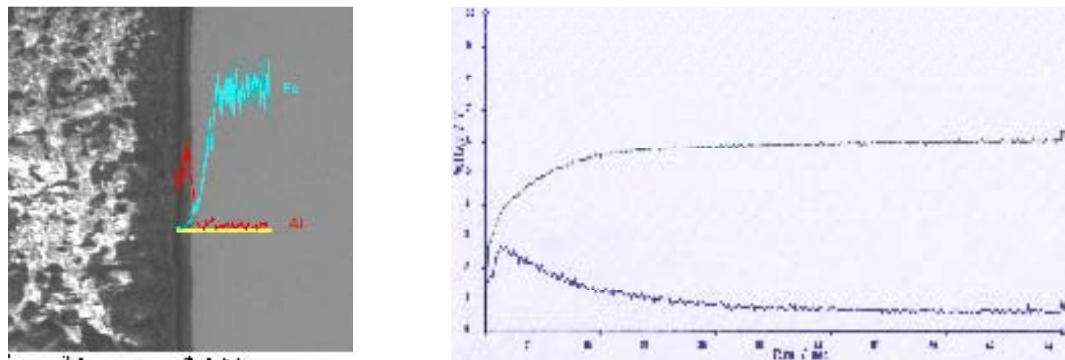
With B samples, treated with the  $AlCl_3$  activator, and C samples, treated with the  $NaCl$  activator, aluminized at  $550^\circ C$  in the period of 4 hours, the change in the concentration of iron and aluminum is not constant throughout the layer, Figures 4 and 5, which indicates that aluminum is dissolved in a stable solution in the iron substrate.



**Figure 3.** Cross- section SEM image and GDOS with the distribution of Al and Fe in a coating formed in an  $NH_4Cl$  activated pack at  $620^\circ C$  for 4 h.



**Figure 4.** Cross- section SEM image and GDOS with the distribution of Al and Fe in a coating formed in an  $AlCl_3$  activated pack at  $550^\circ C$  for 4 h.



**Figure 5.** Cross- section SEM image and GDOS with the distribution of Al and Fe in a coating formed in a  $NaCl$  activated pack at  $550^\circ C$  for 4 h.

## 4 CONCLUSION

A low temperature aluminizing treatment using adequate activators can be applied to hot work steels.

The  $\text{NH}_4\text{C}$  activator provides a high level of activity and the formation of intermetallic connection layers, which makes it more suitable for high temperature aluminizing and for conditions of high oxidation resistance (gas turbine blades). The activators  $\text{AlCl}_3$  and  $\text{NaCl}$  provide a low level of activation and a lower aluminum potential, which results in obtaining aluminum in a stable solution in the surface layer of the substrate.

Low temperature aluminizing is particularly suitable for tool steels previously hardened and tempered at a temperature higher than the temperature of aluminizing. Thus, superior exploitation properties of the base material are preserved.

Based on the above facts, it can be concluded that aluminizing could be applied as a useful technique to improve the surface characteristics of steel components.

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