

## A LOW TEMPERATURE ALUMINIZING TREATMENT OF HOT WORK TOOL STEEL

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### Summary

Conventional aluminizing processes by pack cementation are typically carried out at elevated temperatures. A low temperature powder aluminizing technology was applied to the X40CrMoV5-1 hot tool steel. The aluminizing temperature was from 550 °C 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), and glow discharge optical spectroscopy (GDOS) were applied to observe the cross-sections and the distribution of elements.

*Key words:* low temperature pack aluminizing; aluminium oxide coatings, high pressure die casting, reactive soldering

### 1. Introduction

Die casting is a cost-efficient process for forming parts made of aluminium-, zinc-, magnesium-, and copper-based alloys [1-3]. 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, longer 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 aluminium die casting [4-7]. Numerous past studies have focused mainly on the study of thermal fatigue cracking [8-10]. However, with the development of the die casting industry, increasingly more consideration is being given to die soldering, resulting in a considerable increase in the efficiency and productivity of the die casting process. Soldering, or die sticking, in aluminium die casting occurs during the die casting process when the molten aluminium „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 aluminium 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 aluminium alloy enters the die and destroys the protective film (coating or lubricant) on the die. The molten aluminium then comes into contact with the die surface. The iron in the die substrate dissolves the melt whereas aluminium and other elements in the melt diffuse into the die. As a result, a layer of intermetallic compounds 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 vapour deposition (PACVD), physical vapour deposition (PVD), and duplex treatments combining nitriding with physical vapour deposition (PVD) coatings [11-15]. The literature reveals that alumina layers could be formed on steels containing aluminium, 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 aluminium to form a protective alumina ( $\text{Al}_2\text{O}_3$ ) layer on the surface under high-temperature oxidation conditions [16]. 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 aluminium die casting [17]. Oxidizing the Incoloy MA956 alloy has several advantages over oxidizing the X40CrMoV5-1 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 the X40CrMoV5-1 tool steel, but Incoloy MA956 could replace the X40CrMoV5-1 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 due to microstructure changes associated with grain coarsening and carbide precipitation [14,15]. 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 X40CrMoV5-1 hot work tool steel. From the aluminium-iron phase diagram, Figure 1, we can determine the parameters of low temperature aluminizing which enables the introduction of aluminium into the substrate below the solubility of aluminium 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 aluminium 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 aluminium powder, inert filler, and various activators. Subsequently, oxidizing gas atmosphere containing either  $\text{H}_2\text{O}$  or  $\text{CO}_2$  was applied to oxidize the aluminium dissolved in the surface layer to obtain a compact layer of  $\text{Al}_2\text{O}_3$ , which is thermodynamically stable up to very high temperatures and which cannot be reduced by liquid aluminium.

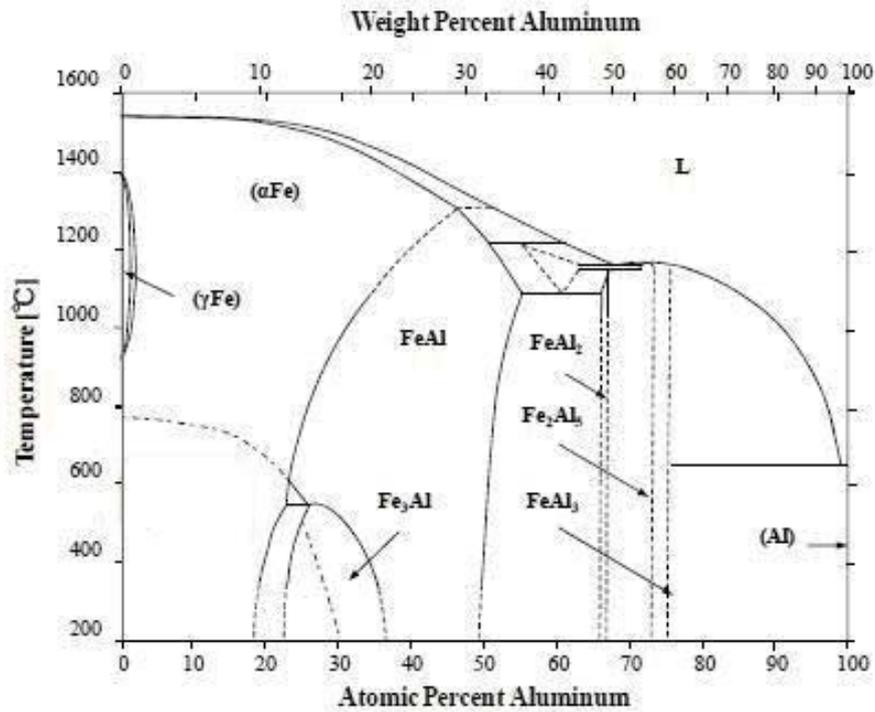


Fig. 1 Binary phase diagram of Al-Fe [18]

## 2. Experimental procedures

The substrate material used for investigation was the X38CrMoV5 1 hot-work steel. The chemical composition of the material, analyzed by using the bulk glow discharge optical spectroscopy, was as follows: 0.35 % C, 1.05 % Si, 5.0 % Cr, 1.3 % Mo, 0.32 %V. Samples were prepared to dimensions of approximately 20 x 40 x 2 mm and then manually polished using the 600 grit SiC abrasive paper and ultrasonically cleaned with acetone.

Aluminizing powder mixtures were prepared by weighing out and mixing appropriate amounts of aluminium 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 (aluminium 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, Figure 2, argon-purged with a flow of 5 l/min and placed into a horizontal 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.

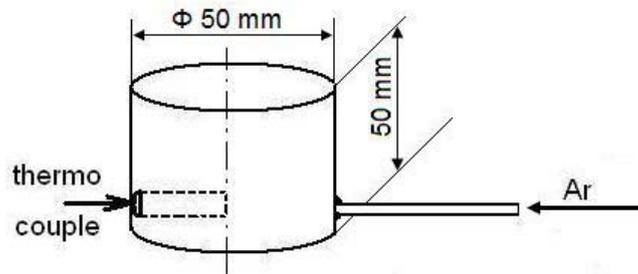


Fig. 2 Schematic diagram of the special retort for powder aluminizing

For metallographic examination, specimens were the 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 220 – 800 grit abrasive papers, while the final polishing was carried out using diamond paste of up to 1  $\mu\text{m}$ . 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 the scanning electron microscopy (SEM) TESCAN VEGA TC with the energy dispersive spectroscopy (EDS) OXFORD INCA and the glow discharge optical spectroscopy (GDOS) LECO SPECTRUMAT- 850A Glow Discharge Optical Spectrometer.

### 3. Results and discussion

With A samples, treated with the  $\text{NH}_4\text{Cl}$  activator, aluminized at 620 °C in the period of 4 hours, a layer of 50  $\mu\text{m}$  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 aluminium, Figure 3.

With B samples, treated with the  $\text{AlCl}_3$  activator, and C samples, treated with the  $\text{NaCl}$  activator, aluminized at 550 °C in the period of 4 hours, the change in the concentration of iron and aluminium is not constant throughout the layer, Figures 4 and 5, which indicates that aluminium is dissolved in a stable solution in the iron substrate. The depth of diffusion of aluminium with the application of the  $\text{AlCl}_3$  and the  $\text{NaCl}$  activator was 20  $\mu\text{m}$ .

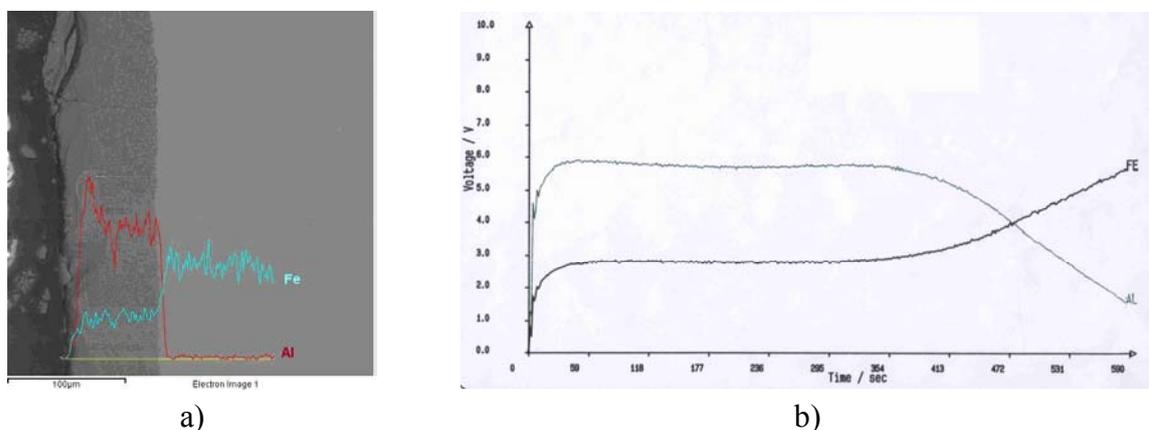
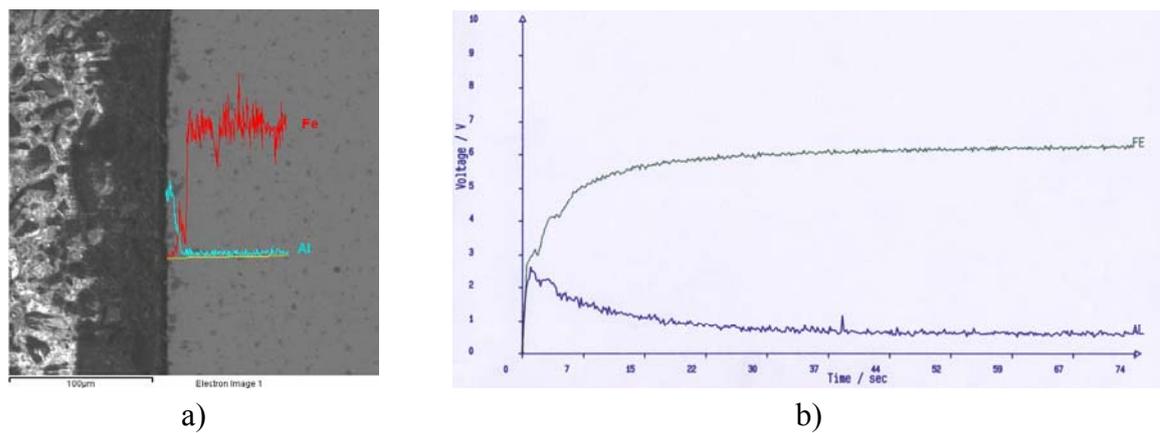
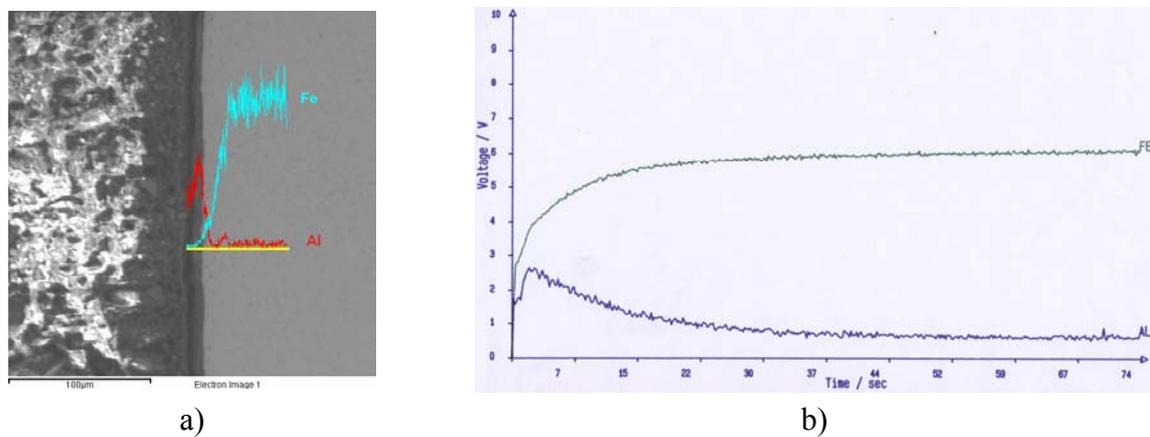


Fig. 3 Distribution of Al and Fe in the coating formed by using the  $\text{NH}_4\text{Cl}$  activator at 620 °C for 4 hours, a/ cross-section SEM image, b/ GDOS



**Fig. 4** Distribution of Al and Fe in the coating formed by using the  $\text{AlCl}_3$  activator at  $550^\circ\text{C}$  for 4 hours, a/ cross-section SEM image, b/ GDOS



**Fig. 5** Distribution of Al and Fe in the coating formed by using the  $\text{NaCl}$  activator at  $550^\circ\text{C}$  for 4 hours, a/ cross-section SEM image, b/ GDOS

#### 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 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 aluminium potential, which results in obtaining aluminium 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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