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Surface state of alloy based on FeAl intermetallic phase matrix after oxidation at 600°C, 700°C and 800°C

Stan powierzchni stopu na osnowie fazy międzymetalicznej FeAl po utlenianiu w temperaturze 600°C, 700°C i 800°C

The aim of the research was to determine the heat resistance of $Fe_{40}Al_5Cr_{0.2}$ TiB alloy based on surface condition analysis. Oxidation was carried out at 600°C, 700°C and 800°C in air. The surface condition was characterized using a scanning electron microscope. EDS X-ray microanalysis was performed. The obtained results prove the high heat resistance of $Fe_{40}Al_5Cr_{0.2}$ TiB alloy in the temperature range of 600–800°C.

Keywords: intermetallic alloy, FeAl, heat resistance

Celem prowadzonych badań było określenie żaroodporności stopu $Fe_{40}AI_5Cr_{0,2}TiB$ na podstawie analizy stanu powierzchni. Utlenianie prowadzono w temperaturze 600°C, 700°C i 800°C na powietrzu. Scharakteryzowano stan powierzchni za pomocą elektronowego mikroskopu skaningowego (SEM) oraz przeprowadzono badania składu chemicznego metodą mikroanalizy rentgenowskiej (EDS). Uzyskane wyniki wykazały wysoką żaroodporność stopu $Fe_{40}AI_5Cr_{0,2}TiB$ w zakresie temperatury 600–800°C.

Słowa kluczowe: stop międzymetaliczny, FeAl, żaroodporność

1. Introduction

Intensive development of material engineering in recent years has allowed the development and production of innovative alloys based on the FeAI intermetallic phase, which are commonly called intermetallics, in a way that enables their practical use [1–3]. Their potential applications include the energy, automotive and aviation industries. The properties of intermetallics are characteristic of metal materials, which feature high mechanical strength and good plastic properties, and for ceramic materials, which feature high heat resistance, corrosion resistance, and brittleness [1, 4]. FeAI phase matrix alloys contain from 36–51% at. Al and are a real alternative to commonly used heat-resistant steels due to [1, 3–6]:

- high resistance to environments containing sulfur and chlorine,
- high heat resistance,
- high resistance carburizing and oxidizing environments (up to 1100°C),
- low density,
- structural stability up to the melting point,
- high specific strength and modulus of elasticity.

The alloy's high corrosion and creep resistance based on the FeAI intermetallic phase results from an ordered structure referred to as B2, which reduces the self-diffusion coefficient, thus increasing the above-mentioned properties [7–10].

Due to their heat-resistant properties, alloys based on the FeAl intermetallic phase are intended for application in high-temperature conditions (above 600°C) and in corrosive environments [7, 8, 11, 12]. In the available literature, there is no information on corrosion resistance analysis at 600–800°C. So far, heat resistance tests have been carried out in the temperature range of 900–1100°C for 20 h, 40 h, 80 h, 100 h and 200 h [6]. Therefore, it seems justified to determine the heat resistance of Fe₄₀Al₅Cr_{0.2}TiB intermetallic alloy in a temperature range of 600–800°C, which will make it possible to determine the morphology of the microstructure in the assumed operating conditions of the material.

2. Research methodology

The test material consisted of 20×10 mm Fe₄₀Al₅Cr_{0.2}TiB alloy samples (Table 1). The samples were annealed in an oven at 600°C, 700°C and 800°C for 168 h (7 days) and cooled in air. The chemical composition of the material as presented in Table 1 has been selected as part of the author's own long-term research program and was verified in operating conditions.

The research included analysis of scale morphology and of the surface condition of $Fe_{40}AI_5Cr_{0.2}$ TiB alloy samples using a HITACHI S-4200 scanning electron microscope and EDS (Energy Dispersive Spectroscopy) X-ray microanalysis.

Table 1. Chemical composition of Fe ₄₀ Al ₅ Cr _{0.2} TiB alloy
Tabela 1. Skład chemiczny stopu Fe ₄₀ Al ₅ Cr _{0,2} TiB

Alloy / Stop	Chemical analysis, mass % / Skład chemiczny, % mas.				
	Fe	AI	Cr	Ti	В
$Fe_{40}Al_5Cr_{0.2}TiB$	68.22	23.69	5.69	0.19	0.015

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Fig. 1. Surface morphology of the oxide scale formed on $Fe_{40}Al_5Cr_{0.2}TiB$ alloy after annealing at: a) 600°C, b) 700°C, c) 800°C for 168 h Rys. 1. Morfologia powierzchni zgorzeliny stopu $Fe_{40}Al_5Cr_{0.2}TiB$ po wygrzewaniu w temperaturze: a) 600°C, b) 700°C, c) 800°C przez 168 h



Fig. 2. Surface of the oxide scale formed on Fe₄₀Al₅Cr_{0.2}TiB alloy oxidized at 600°C for 168 h (a), results of EDS microanalysis in area 1 (b), results of EDS microanalysis in area 2 (c)

Rys. 2. Powierzchnia zgorzeliny stopu Fe₄₀Al₅Cr_{0.2}TiB po utlenianiu w temperaturze 600°C przez 168 h (a), analiza EDS w miejscu 1 (b), analiza EDS w miejscu 2 (c)

3. Experimental research results

3.1. Morphology of the alloy surface after oxidation

Fig. 1 shows the morphology of the scale surface after oxidation of $Fe_{40}AI_5Cr_{0.2}TiB$ alloy at different temperatures for 168 h. Depending on the applied annealing temperature of the alloy, structure differentiation was observed.

3.2. Chemical composition of corrosion products

After oxidation at 600°C, the surface of the oxidized layer (Fig. 1a) was relatively small compared to the surface of the scale formed at 700°C and 800°C, which was covered with oxides in the form of fine needles (Fig. 5). EDS analysis of the sample after annealing at 600°C (Fig. 2), showed that the surface of the sample is free of oxidation products such as aluminum oxides (Fig. 2a), because the chemical composition does not correspond to the Al₂O₃ phase. The EDS study confirmed the presence of iron and aluminum (Fig. 2b), which are the main components of Fe₄₀Al₅Cr_{0.2}TiB alloy, and chromium, which is added to reduce brittleness at high temperatures. EDS analysis also showed the formation of silicon carbides (Fig. 2c). Silicon does not belong to the chemical composition of Fe₄₀Al₅Cr_{0.2}TiB alloy, however, it may be an impurity resulting from casting the alloy into an ingot containing silicon in its composition. Small amounts of

carbon are added to the alloy while increasing its strength by precipitation hardening of the parent phase. At 700°C, the initial phase of scale formation consisting of Al_2O_3 was found to occur (Fig. 1b).

Examination of the chemical composition by the EDS method samples after annealing at 700°C (Fig. 3) confirmed the formation of a passive Al_2O_3 layer (Fig. 3c). Moreover, Fig. 3b shows that in the area marked as 1 of the EDS analysis (Fig. 3a), constituting the base of the $Fe_{40}Al_5Cr_{0.2}$ TiB alloy, oxygen appears. This may suggest progressing oxidation of the sample from a temperature of 700°C. The formation of a protective Al_2O_3 layer on the surface of the intermetallic alloy $Fe_{40}Al_5Cr_{0.2}$ TiB during high-temperature processes in an oxidizing atmosphere prevents the degradation of the metallic core, thus it is the basic determinant of high heat resistance of the alloy in an air atmosphere at high temperature.

The surface of the sample annealed at 800°C (Fig. 1c) shows clear changes in the surface morphology compared to samples annealed at 600°C and 700°C. The structural basis of the material is heterogeneous and is covered with an unevenly distributed passive layer. Examination of the chemical composition of the sample using the EDS method allows us to determine the presence of oxidation products in the form of Al_2O_3 (Fig. 4b). The surface of the scale formed at 800°C covers a significant part of the native material (Fig. 1c), and the resulting oxides are characterized by



Fig. 3. Surface of oxide scale formed on Fe₄₀Al₅Cr_{0.2}TiB alloy oxidized at 700°C for 168 h (a), results of EDS microanalysis in area 1 (b), results of EDS microanalysis in area 2 (c)

Rys. 3. Powierzchnia zgorzeliny stopu Fe40AlsCr0.2TiB po utlenianiu w temperaturze 700°C przez 168 h, analiza EDS w miejscu 1 (b), analiza EDS w miejscu 2 (c)



Fig. 4. Surface of the oxide scale formed on Fe₄₀Al₅Cr_{0.2}TiB alloy oxidized at 800°C for 168 h (a), results of EDS microanalysis in area 1 (b), results of EDS microanalysis in area 2 (c)





Fig. 5. Morphology of the scale covering $Fe_{40}AI_5Cr_{0.2}TiB$ alloy after oxidation at 800°C for 168 h

Rys. 5. Morfologia zgorzeliny pokrywającej stop $Fe_{40}AI_5Cr_{0,2}TiB$ po utlenianiu w temperaturze 800°C przez 168 h

fine needles (Fig. 5). According to the literature [13], the size of the oxide needles formed depends on the rate of the oxidation process. Based on Fig. 1c, it can be inferred that the scale peels at 800°C, which may be due to stresses arising as a result of scale growth. In the tested temperature range, the greatest changes in scale morphology occur during oxidation at 800°C. On the basis of the performed tests, it can also be concluded that the "limiting point" of the developing oxidation process, which is characterized by the formation of a passive oxide layer, is the temperature of 700°C.

4. Summary

Based on surface condition tests of $Fe_{40}Al_5Cr_{0.2}TiB$ alloy during oxidation in the temperature range of 600–800°C for 168 h (7 days) and an analysis of literature concerning alloys based on the FeAl intermetallic phase, the following conclusions can be drawn:

1. The processes related to the formation of a protective oxide layer do not occur significantly at 600°C. The scale surface at the above temperature is smooth compared to 700°C and 800°C.

- 2. The formation of the Al_2O_3 scale begins at a temperature lower than 700°C, while intensification of the oxidation process occurs in the range of 700–800°C. Further research should focus on a precise analysis of this range in order to determine the exact temperature at which the oxidation process starts.
- 3. The surface of the alloy at 800°C is covered with needle-like alumina scale. The structure of oxides is influenced by the speed of the oxidation process.
- 4. Due to the stresses occurring as a result of scale growth or as a result of thermal stresses arising after the cooling of $Fe_{40}AI_5Cr_{0.2}TiB$ alloy, the resulting scale starts to flake (peel off).
- 5. The conducted tests show that the heat resistance of the $Fe_{40}AI_5Cr_{0.2}TiB$ intermetallic alloy in an oxidizing environment up to 700°C is high because there is no degradation of the structure caused by high-temperature oxidation.

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