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## COMPARISON OF HIGH TEMPERATURE CORROSION RESISTANCE IN GASEOUS ENVIRONMENT OF ALLOYS BASED ON INTERMETALLIC PHASE MATRIX Fe<sub>40</sub>Al<sub>5</sub>CrZrB AND STEEL X12CrCoNi2120

### PORÓWNANIE WYSOKOTEMPERATUROWEJ ODPORNOŚCI KOROZYJNEJ W ŚRODOWISKU GAZOWYM STOPÓW NA OSNOWIE FAZY MIĘDZYMETALICZNEJ Fe<sub>40</sub>Al<sub>5</sub>CrTiB I STALI X12CrCoNi2120

The paper presents results of the research which aim was to compare the corrosion resistance of alloys based on intermetallic phase after Fe<sub>40</sub>Al<sub>5</sub>CrTiB crystallization and after forming the steel corrosion resistant X12CrCoNi2120 species. The tests were performed for steel at temperature of 700°C Fe<sub>40</sub>Al<sub>5</sub>CrTiB and for alloy at 1100°C and 9% O<sub>2</sub> 0.2% HCl + SO<sub>2</sub> + 0.08 N<sub>2</sub> environment. In the research the changes of weight after corrosion tests, observations of the surface, specified chemical and phase composition of corrosion products were made. The obtained results of the study showed a very good corrosion resistance of Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloys in high temperature and environments containing oxygen, sulfur and chlorine as compared to the corrosion resistance of the steel grade X12CrCoNi2120. Results of the research conducted in this scope are the basis for further research.

*Keywords:* corrosion resistance, high-temperature corrosion, alloys intermetallic FeAl matrix phase

W artykule przedstawiono wyniki badań mających na celu porównanie odporności korozyjnej stopów na osnowie fazy międzymetalicznej Fe<sub>40</sub>Al<sub>5</sub>CrTiB po krystalizacji i po przeróbce plastycznej ze stałą odporną na korozję gatunku X12CrCoNi2120. Badania wykonano dla stali w temperaturze 700°C oraz dla stopu Fe<sub>40</sub>Al<sub>5</sub>CrTiB w temperaturze 1100°C w środowisku: 9% O<sub>2</sub>+0,2 HCl + 0,08% SO<sub>2</sub>+ N<sub>2</sub>. Określono zmiany masy po badaniach korozyjnych, wykonano obserwacje stanu powierzchni, określono skład chemiczny i fazowy produktów korozji. Uzyskane rezultaty badań wykazały bardzo dobrą odporność stopów Fe<sub>40</sub>Al<sub>5</sub>CrTiB na korozję wysokotemperaturową w środowisku zawierającym tlen, siarkę i chlor w porównaniu do odporności korozyjnej stali gatunku X12CrCoNi2120. Wyniki badań prowadzonych w tym zakresie stanowią podstawę do dalszych prac badawczych.

## 1. Introduction

Fe-Al alloys are used in many industries. They are used in the automotive, energy, petrochemical, aerospace or production equipment operating at elevated temperatures [1-2]. Such a broad spectrum of applications, is due to the fact that they have a specific combination of resistance to an oxidizing atmosphere up to 1100°C, especially in environment containing sulfur and chlorine, and a low density and strength at elevated temperatures [3-6]. Alloys based on FeAl matrix phase due to a higher aluminum content, and thus a lower density and a relatively low price of material (compared to the price of stainless steels and steel, which contain expensive elements such as chromium, nickel and molybdenum) are the subject of a wide interest [5]. The disadvantages include the low ductility at ambient temperature and the decrease in strength at temperatures above 700°C, resulting from the tendency of these materials to the coarseness of the crystallization process [7-9]. However, there are methods which aim is increasing the strength

of these alloys, and overcoming the problems associated with poor fracture toughness at room temperature [10-13].

The paper presents the results of corrosion resistance of the Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy after crystallization and plastic forming, in an environment containing oxygen, sulfur and chlorine. Comparison of corrosion resistance of high-temperature Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy and corrosion resistance of X12CrCoNi2120 steel was made. The change of mass caused by corrosive phenomena in relation to the surface of the samples was defined. The study was completed at the point of determining the state of the surface chemical composition, microanalysis of corrosion products, and their X-ray phase analysis.

## 2. Materials and Experimental procedure

Material used in the research consisted of Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy samples after crystallization and after forming and samples with corrosion-resistant steel X12CrCoNi2120 species.

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The chemical composition of the alloy is shown in Table 1. Plastically formed material was obtained by extrusion method, which is the authors own technology and is protected by the patent. This method hasn't been used in other technological solutions. Corrosion resistance tests were carried out at 700°C for steel and 1100°C for the alloy in the environment  $9\% \text{ O}_2 + 0.2\% \text{ HCl} + 0.08\% \text{ SO}_2 + \text{N}_2$ . Testing time of trial was 20h, 40h, 60h, 80h and 100 h. For each sample the weight before and after the corrosion test was checked on laboratory weight accuracy  $10^{-4}\text{g}$ . Surface of the samples for which weight change was determined equaled  $10 \text{ mm}^2$ . Alloy microstructure based on intermetallic phase FeAl before corrosion tests was analyzed by light microscope OLYMPUS GX51. Determination of the corrosion surface was carried out on a scanning electron microscope equipped with a Hitachi S4200 ray detector EDS (Energy dispersive spectroscopy), which allowed to specify chemical composition of the corrosion products. Phase composition was determined by X-ray phase analysis using X-ray Jeol JDX 7-S diffractometer.

TABLE 1

Assumed and real chemical composition of the Fe40Al5CrZrB alloy

Fe40Al5CrZrB Alloy	Content of element [% mas.]				
Element	Fe	Al	Cr	Ti	B
Assumed	70,1	24,5	4,9	0,11	0,02
Real	70,1	24,5	5,3	0,126	0,014

### 3. Results

Analyzing the measurement results after corrosion test for FeAl alloy and X12CrCoNi2120 steel, it was concluded that the change of samples weight occurred in different degrees for various materials (Fig. 1-2). For samples of Fe40Al5CrTiB alloy small change of mass was observed. Corrosion resistance of X12CrCoNi2120 steel (Fig. 1) (used to produce the exhaust valves of internal combustion engines) is much lower in comparison to the corrosion resistance of an alloy on the intermetallic matrix Fe40Al5CrTiB (Fig. 2). FeAl alloy after crystallization has lower corrosion resistance as compared to the corrosion resistance of FeAl alloy processed plastically. This may be due to the fact that the original FeAl alloy microstructure has a number of disadvantages, such as: shrinkage porosity, dendrites, heterogeneity and coarseness of the chemical composition of the material (Fig. 3-4). Alloys on the FeAl intermetallic phases are particularly vulnerable to the above-mentioned casting defects [4-6]. Such phenomenon causes a substantial limitation for the practical use of these materials in the slate directly after the primary crystallization. Plastic processing of FeAl alloys with intermetallic matrix largely removes the casting defects (Fig. 5-6). Applying plastic processing resulted in considerable breaking-up of microstructure and removing shrinkage and voids, thus led to the improvement of physico-chemical and mechanical properties, and consequently to the improvement of corrosion resistance. The EDS X-ray microanalysis of chemical composition of FeAl samples shows that in the oxidation process corrosion products are formed mainly containing Fe compounds,

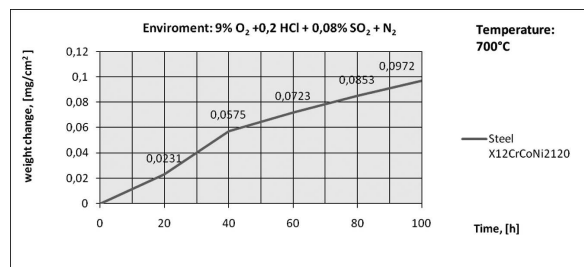


Fig. 1. Mass change as a function of time for the corrosion test of X12CrCoNi2120 steel

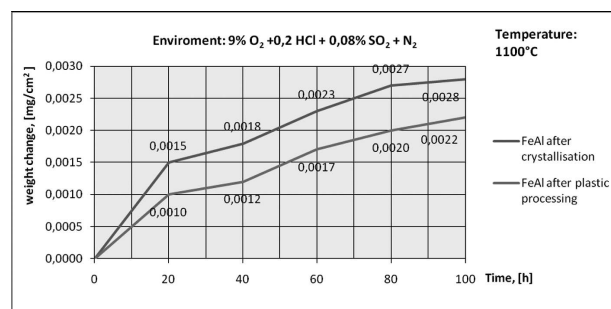


Fig. 2. Mass change as a function of time for the corrosion test of Fe40Al5CrTiB steel for the alloy after crystallization and plastic processing

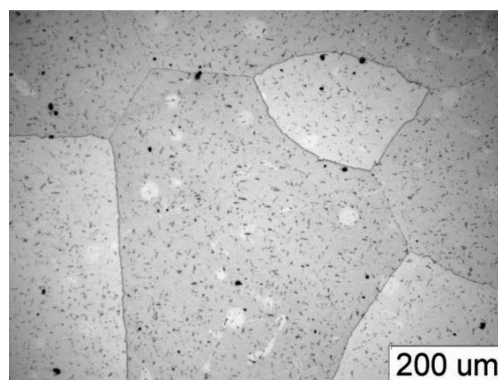


Fig. 3. Microstructure of the Fe40Al5CrTiB alloy after primary crystallization

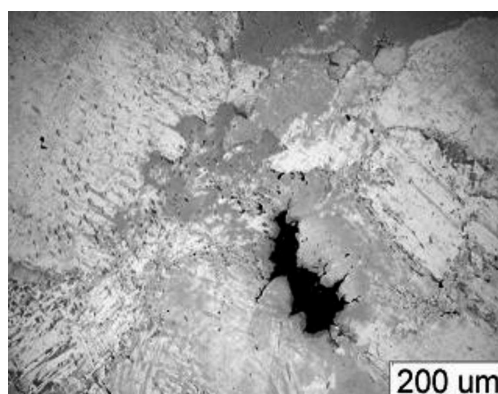


Fig. 4. Microstructure of the Fe40Al5CrTiB alloy after primary crystallization, visible coarseness and voids

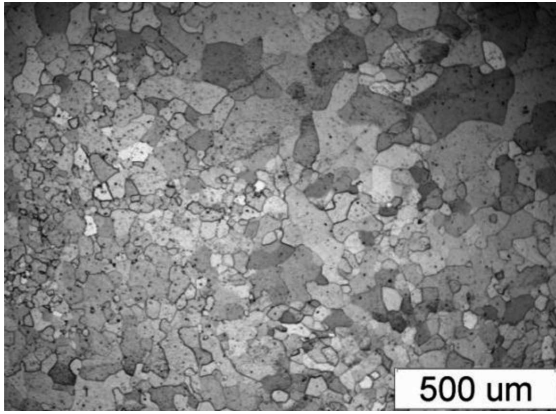


Fig. 5. Microstructure of Fe40Al5CrTiB alloy after plastic processing by means of extruding

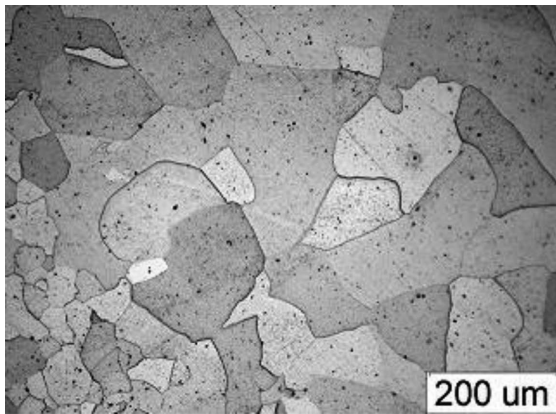


Fig. 6. Microstructure of Fe40Al5CrTiB alloy after plastic processing, visible irregular size of particle and separations

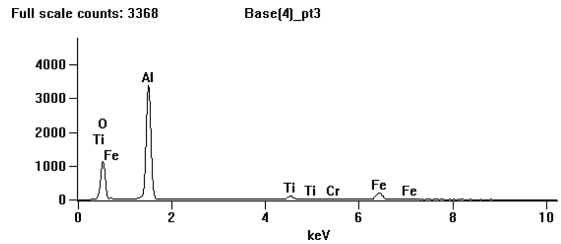
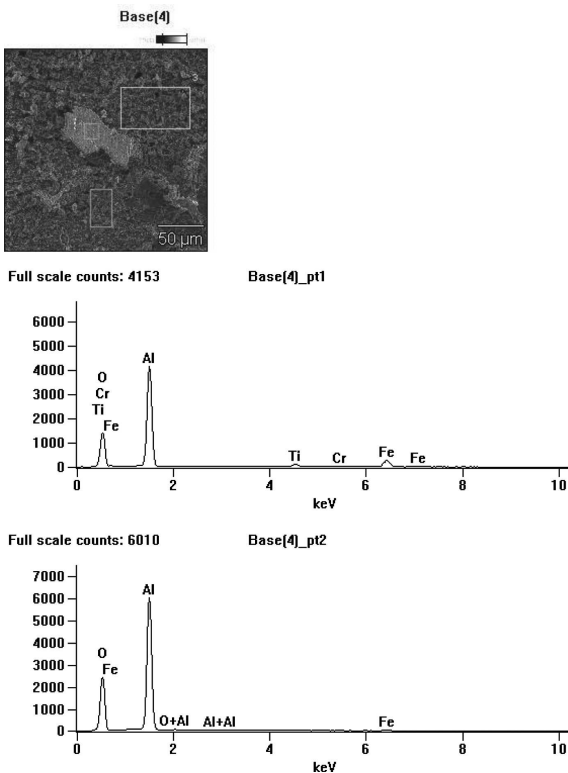


Fig. 7. Microanalysis of chemical composition of Fe40Al5CrTiB alloy after crystallization and corrosion test

resulting in a loss of iron from the surface of the material (Fig. 7-8). Observations of the samples surface made by a scanning electron microscope revealed that the surface areas were aluminum-containing oxides. The presence of  $Al_2O_3$  passive layers gave FeAl alloy very good heat resistance in an oxidizing environment. Presumably, in the first stage of corrosion process the iron was oxidised at the surface layer. There was also heat-resistant layer of  $Al_2O_3$ , which hermetically isolate the rest of the material from oxygen and sulfur (Fig. 9-10). X-ray phase analysis of corrosion products only showed the presence of  $Al_2O_3$  (Fig. 11) obtained aluminum oxide occurred in two allotropic forms.

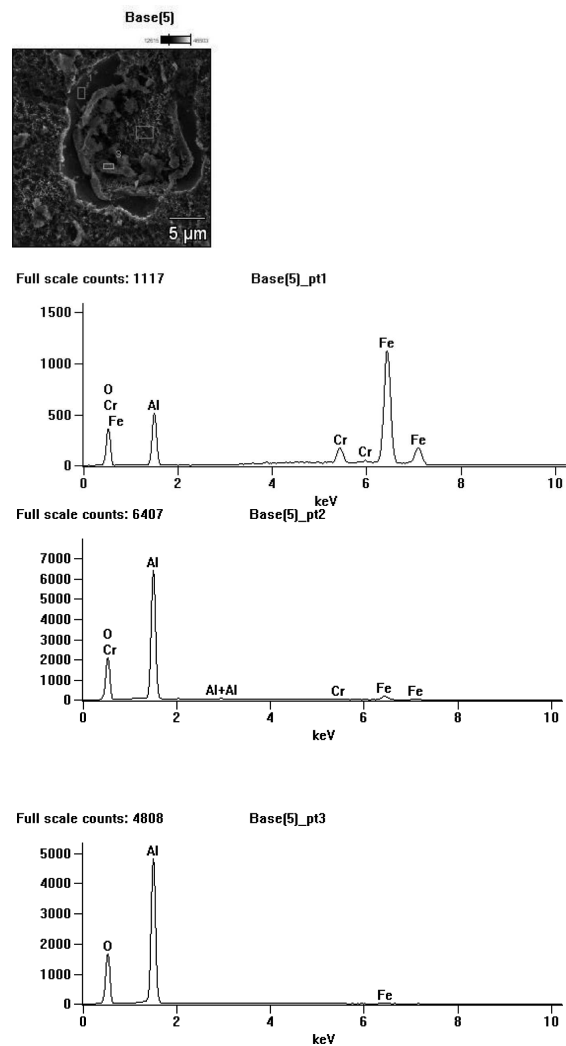


Fig. 8. Microanalysis of chemical composition of Fe40Al5CrTiB alloy after crystallization and corrosion test



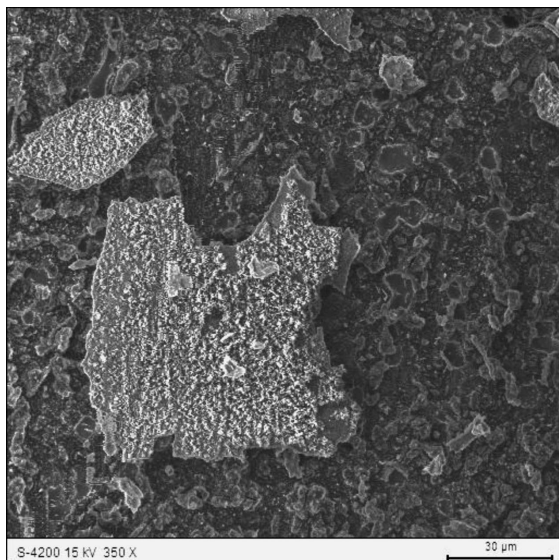


Fig. 9. Structure of the crystallized Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy after corrosion tests, scanning electron microscope

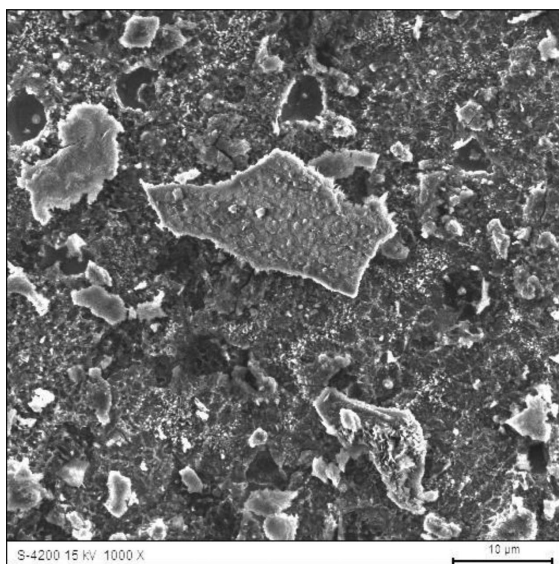


Fig. 10. Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy structure after plastic processing, the corrosion tests, scanning electron microscope

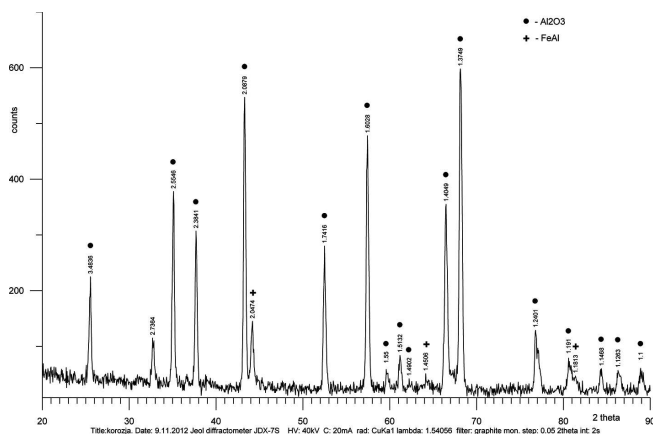


Fig. 11. Results of X-ray phase analysis of the Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy corrosion products

#### 4. Conclusions

Research and analysis of the results lead to the conclusion that the test materials show different high temperature corrosion resistance. Composition based on Fe<sub>40</sub>Al<sub>5</sub>CrTiB intermetallic phase has a much higher corrosion resistance than the corrosion resistance of X12CrCoNi2120 steel despite of the fact that the test for steel was conducted at 700°C, the temperature at which elements of that steel grade work and for the Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy experiment temperature was 1100°C. A very good corrosion resistance of Fe<sub>40</sub>Al<sub>5</sub>CrTiB alloy is ensured by passive layer of Al<sub>2</sub>O<sub>3</sub> formed on its surface. Phenomena of high-temperature corrosion of FeAl intermetallic matrix alloy require further research.

#### Acknowledgements

This research was financed partially within the framework of the Scientific Research Project No. 2188/B/T02/2011/40 heated by dr inż. Janusz Cebulski.

#### REFERENCES

- [1] <http://suw.biblos.pk.edu.pl/resources/i1/i5/i5/i2/r1552/> AugustynPieniazekJ\_SkladFazowy.pdf
- [2] Z. Bojarski, W. Przetakiewicz, *Materiały metalowe z udziałem faz międzymetalicznych*. Warszawa, BEL Studio, 89 (2006).
- [3] J. Cebulski, S. Lalik, R. Michalik, Ocena odporności korozyjnej stopów na osnowie fazy międzymetalicznej FeAl o zawartości aluminium 38-42%at., *Ochrona przed korozją* **2**, 62 (2007).
- [4] [http://ksiegarnia.iknt.pl/uploads/files/Materiały\\_metalowe\\_fragment.pdf](http://ksiegarnia.iknt.pl/uploads/files/Materiały_metalowe_fragment.pdf)
- [5] J. Xia, C.X. Li, H. Dong, Thermal oxidation treatment of B2 iron aluminide for improved wear resistance, *Wear* **258**, 1804-1812 (2005).
- [6] W.Z. Zhu, S.C. Deevi, Dispersion of iron aluminide with polyelectrolyte in water, *Materials Research Bulletin* **38**, 1227-1233 (2003).
- [7] J. Cebulski, J. Barcik, Plasticity of alloy based on the matrix of intermetallic FeAl phase, *Archives of Metallurgy* **45**, 315-329 (2000).
- [8] M. Palm, G. Sauthoff, Deformation behaviour and oxidation resistance of single-phase and two-phase L21-ordered Fe-Al-Ti alloys, *Intermetallics*, **12**, 1345-1359 (2004).
- [9] T. Grosdidier, J.X. Zou, N. Stein, C. Boulanger, S.Z. Hao, C. Dong, Texture modification, grain refinement and improved hardness/corrosion balance of a FeAl alloy by pulsed electron beam surface treatment in the "heating mode", *Scripta Materialia*, **58**, 1058-1061 (2008).
- [10] A. Kazmierczak-Balata, J. Bodzenta, P. Szperlich, K. Wokulska, J. Kucytowski, T. Lukasiewicz, W. Hofman, Determination of thermal, elastic, optical and lattice parameters of GdCOB single crystals doped with Nd<sup>3+</sup> and Yb<sup>3+</sup> ions *Journal of Alloys and Compounds* **481**(1-2), 622-627 (2009).
- [11] L. Yuan, H.M. Wang, Hot corrosion behaviors of a Cr<sub>13</sub>Ni<sub>5</sub>Si<sub>2</sub>-based metal silicide alloy in Na<sub>2</sub>SO<sub>4</sub>+ 25 wt.% K<sub>2</sub>SO<sub>4</sub> and Na<sub>2</sub>SO<sub>4</sub>+ 25wt.% NaCl molten salts, *Intermetallics*, **18**, 324-329 (2010).
- [12] H. Shi, D. Guo, Y. Ouyang, Structural evolution of mechanically alloyed nanocrystalline FeAl intermetallics, *Journal of Alloys and Compounds*, 207-209 (2008).
- [13] J. Cebulski, S. Lalik, Changes in the structure of Allom on the matrix of FeAl intermetallic phase after primary crystallization and homogenizing treatment, *Journal of Materials Processing Technology*, 4-8 (2005).