THE EFFECT OF PHASE COMPOSITION AND DISTRIBUTION ON COEFFICIENT THERMAL EXPANSION OF FE_3AL BASED IRON ALUMINIDES

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Abstract

The iron aluminides can be applied in many engineering applications due to the wide range of their positive properties. One possibility of their use could be for machine components working at elevated temperatures in corrosive environments. But it is necessary to know one important characteristic for use iron aluminides in high-temperature applications - their thermal expansion.

Generally, the high-temperature properties are affected by alloys structure mainly by second phases distribution. From this point of view the same effect for coefficients of thermal expansion (CTE) could be supposed. CTE of representative Fe_3Al - type iron aluminides with different structure type was studied in temperature range 340 - 1200 °C. The main aim of this paper is to find connections between the structure of alloys and CTE.

Keywords: Coefficient of thermal expansion (CTE), Fe_3Al - type iron aluminides, HT- dilatation

1. INTRODUCTION

The iron aluminides are one of the most promising materials with many advantages, which predetermine them for use in structural applications. The main advantages of iron aluminides are good corrosion resistance, even at elevated temperatures, high strength at room temperature, lower specific weight in comparison with stainless steels and relatively low cost of raw materials. However, iron aluminides have some disadvantages (for example a sharp drop in strength above 600 °C or limited ductility at room temperature), which have impeded their mass extension [1-3]. One possible way how to eliminate these negative characteristics is to alloy the binary alloy by elements such as for example Nb, Zr or Cr [4-8].

The knowledge of behavior at high temperatures is necessary for the use of iron aluminides in high-temperature applications. The measurement of their coefficient of thermal expansion is important because CTE together with the high-temperature strength limits the use of these materials [9].

It was shown that high-temperature properties of zirconium dopped iron aluminides are affected by the second phase distribution [10]. The same effect for CTE could be supposed.

The methods for measuring of thermal expansion can be divided into two basic groups - the methods of absolute and relative measurement. The direct measurement of dilatation, X-ray method and interference method are the most frequently used methods from the group of absolute measurement methods. Among the relative methods of dilatation measurement belongs the tube-method, interference or strain-gauge method [11].

The dilatometer-machines can be divided into horizontal and vertical types according to the structural design. Both types of machines have advantages and disadvantages. The horizontal dilatometers have minimal thermal effect on the sample itself. The disadvantage of this arrangement is the friction that occurs between the sample and the holder [11-12].

The medium coefficient α_{med} is determined from the relationship (1):

$$\alpha_{med} = \frac{l_T - l_0}{T - T_0} \frac{1}{l_0} \quad (1)$$

l_T - length of the sample at the given temperature; l_0 - length of the sample at the room temperature (20 °C)
T - given applied temperature; T_0 - room temperature (20 °C)

Recently expansion tests of iron aluminides [13-14] showed, that these materials exhibit the same expansion as steel up to 800 °C. Greater expansion of iron aluminides can be observed above this temperature. FeAl - type alloys exhibit about 8 % higher expansion than Fe_3Al - type alloys.

2. MATERIALS AND EXPERIMENTAL METHODS

All iron aluminide alloys were prepared by vacuum induction melting and casting. Ingots were annealed at 1200 °C for 2 hours and then rolled to the final thickness at 1200 °C in several steps with 15 % thickness reduction in one pass. The nominal chemical composition of the investigated samples is given in **Table 1**. For dilatation measuring the cylindrical or cuboidal specimens (of approximately 15 mm) were cut on the **Table 1**.

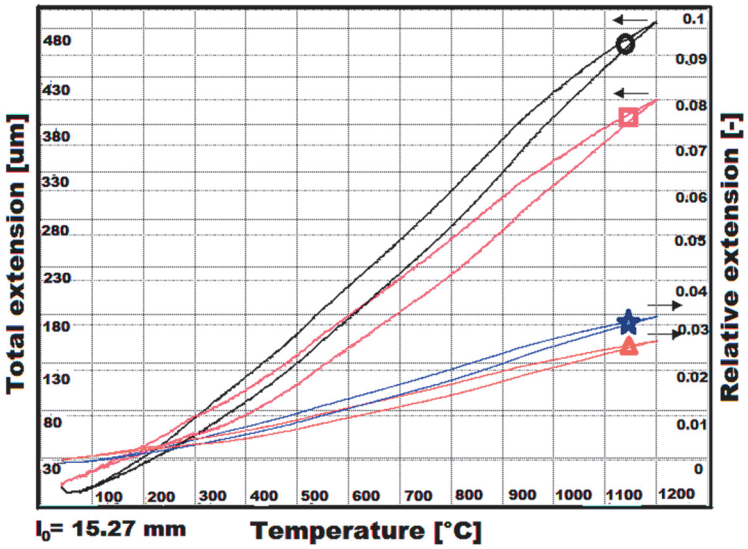
Table 1 The nominal chemical composition of the samples (at.%)

Sample	Fe	Al	Cr	Nb	Zr	C
Fe_3Al-CrNbC	balance	28.1	5.0	0.5	-	0.2
Fe_3Al-CrZrC	balance	29.7	3.7	-	0.3	0.2
Fe_3Al-Nb as received	balance	27.3	-	4.77	-	0.05
Fe_3Al-Nb 1200 °C/24h	balance	27.3	-	4.77	-	0.05

Table 2 The applied temperature cycle for dilatation measurement

step	conditions
1	25 - 250 °C → heating rate 7 °C / min
2	250 - 1200 °C → heating rate 4 °C / min
3	1200 °C → delay at temperature 15 min
4	1200 - 700 °C → cooling rate 4 °C / min
5	700 - 25 °C → cooled at a rate less than 4 °C / min

Fig. 1 Expansion curve of the Fe_3Al - CrZrC



green curve (circle) - the total extension of (sample + sapphire adapter rod)
$dl = D_{total}$ [mm];
pink curve (square) - the absolute extension of sample;
$dl_{revisionary} = D_{total} - D_{sapphire}$ [mm];
blue curve (star) - the relative extension of the system; $r = dl / l_0 \cdot 100$ [%]
red curve (triangle) - the relative extension of the sample;
$r_{revisionary} = dl_{revisionary} / l_0 \cdot 100$ [%]

precise saw Isomet 1000. Thermal expansion was measured by means of horizontal dilatometer. Applied temperature

cycle for dilatation measurement is given in **Table 2**. Repeatedly reached deviation was from 5 to 7 % in the temperature range 25 - 600 °C, 2 - 4% in range 600 - 1000 °C and 0.5 - 1 % in range 1000 - 1200 °C. The example of registered expansion curve is shown in **Fig. 1**. The microstructure was observed after oxide-polishing by suspension OP-S by the light optical microscope (LOM) Nikon Epiphot 200 (with use Differential interference contrast - DIC). The microstructural details and the phase composition were studied by the scanning electron microscope (SEM) Tescan Vega XMU equipped by Bruker detector for energy dispersive analysis (EDX).

3. RESULTS AND DISCUSSION

3.1 Microstructure and phase identification

The matrix of Fe_3Al-CrNbC alloy (see **Fig. 2**) is very coarse-grained with the grains dimension in order of hundreds micrometers. In the matrix there is regularly distributed a large number of precipitates with rounded shape and with dimensions approximately 15 - 20 μm and then very fine particles with dimensions in order of units micrometers. The chemical composition of matrix and particles was verified by EDX. All precipitates were identified as NbC. Chromium was completely dissolved in the matrix. It is consistent with Fe-Al-Cr ternary diagram [15]. The chromium content in the matrix was around 4.7 at.% (it was verified by EDX). More details about the structure of Fe_3Al-CrNbC is in [16].

The matrix of Fe_3Al-CrZrC alloy (see **Fig. 3**) is the same type as the Fe_3Al-CrNbC matrix. It was very coarse-grained with the grains dimension in order of hundreds micrometers. There were observed particles of Laves phase (some of particles with ZrC core - confirmed by EDX) in the structure of Fe3Al-CrZrC. These particles are present on grain boundaries and also inside the grains, however they were distributed inhomogeneously and sometimes in clusters.

At high content of niobium there formed eutectic areas in the structure of material Fe3Al-Nb as received (**Fig. 4**). These areas are a mixture of α (Fe,Al) and $(Fe,Al)_2Nb$ Laves phase probably (according to [17, 18]). Laves phase is present in eutectic areas as globular or elongated particles (size 1-15 μm). The chemical composition of the matrix and precipitates was verified by EDX analysis. Average chemical composition of the particles was determined Fe-(18.5±1.6)Al-(25.1±1.1)Nb.

The structure of material Fe_3Al-Nb 1200 °C/24 h (after annealing 1200 °C/24 h) is shown in **Fig. 5**. Eutectic areas are partly dissolved after annealing, but some of remaining secondary phase particles $(Fe,Al)_2Nb$ got coarse up to 50 μm. The average chemical composition of these particles was determined (by EDX analysis): Fe-(16.1±0.6)Al-(26.6±0.9)Nb. But the particles with twofold content of niobium (average chemical composition Fe-(22.1±0.5)Al-(49.2±2.3)Nb) were found in the matrix too. These particles formed probably due to an excess of niobium in the matrix, which comes from the dissolved eutectic. According to [17,18] it could be either μ-phase with a stoichiometric formula $Nb_{19}(Fe_{1-x}Al_x)_{21}$ or μ'-phase Nb_2FeAl.

3.2 Coefficient of thermal expansion (CTE)

The values of CTE for investigated alloys were measured by means of the horizontal dilatometer and the curves of temperature dependence on the CTE are shown in **Fig. 6**.

CTE values of the alloy Fe_3Al-CrNbC are lower at all investigated temperatures in comparison to Fe_3Al-CrZrC (**Fig. 6**.) This phenomenon may be caused by higher content of chromium dissolved into the matrix that is solidified by solid solution. Also the size and more homogeneous distribution of the secondary phase in the Fe_3Al-CrNbC alloy could have beneficial effect on the CTE values.

The annealing 24 h at 1200 °C also has a significant influence on CTE values (compared with Fe_3Al-Nb, see **Fig. 6**). CTE values of the sample Fe_3Al-Nb as received are lower than CTE values of annealed sample Fe_3Al-Nb 1200 °C/24 h. However, the CTE values for Fe_3Al-Nb as received show significant increase

with increasing temperature. This phenomenon may be caused by dissolution of the eutectic areas, which hampered the expansion of the material at lower temperatures. The eutectic areas could have similar effect as reinforcing phase in composites. The CTE values for annealed sample Fe_3Al-Nb 1200 °C/24 h are relatively stable from medium to high temperatures (the values fluctuate between $19 \cdot 10^{-6}$ and $20.5 \cdot 10^{-6}$ K^{-1}). It is required for temperature depended dimension changes to vary in the minimal possible interval within a wide temperature range.

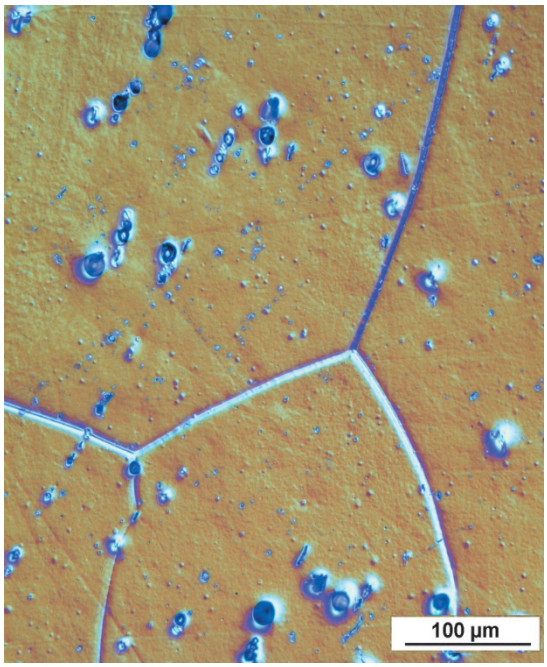


Fig. 2 The structure of the Fe_3Al - CrNbC (LOM)

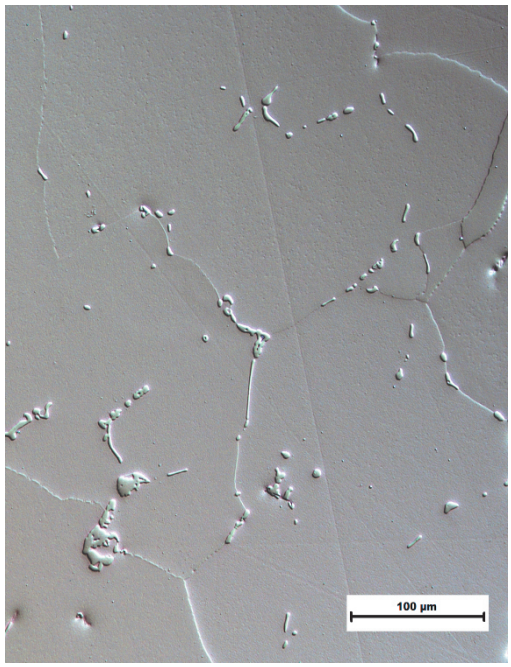


Fig. 3 The structure of the Fe_3Al - CrZrC (LOM)

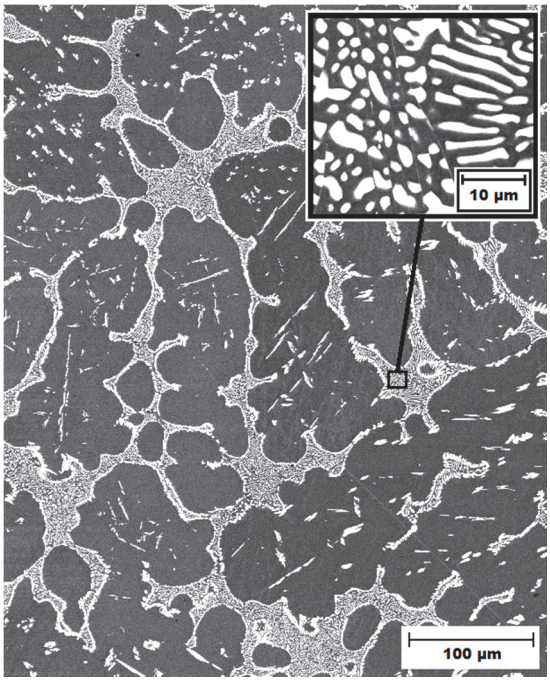


Fig. 4 The structure of the Fe_3Al-Nb as received (BSE)

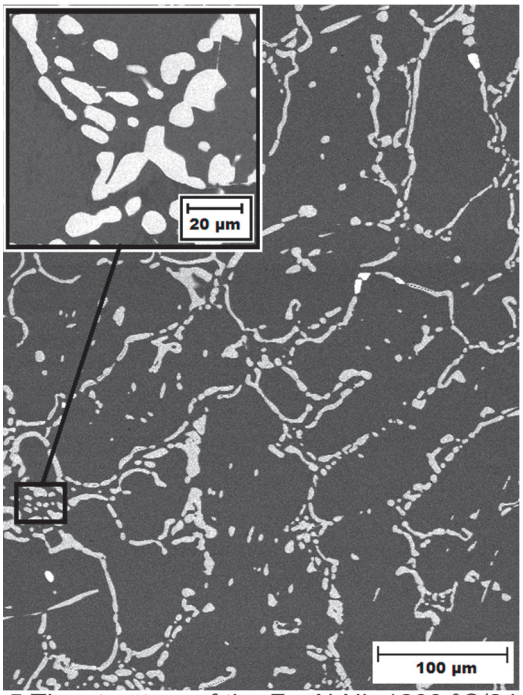


Fig. 5 The structure of the Fe_3Al-Nb 1200 °C/24 h (BSE)

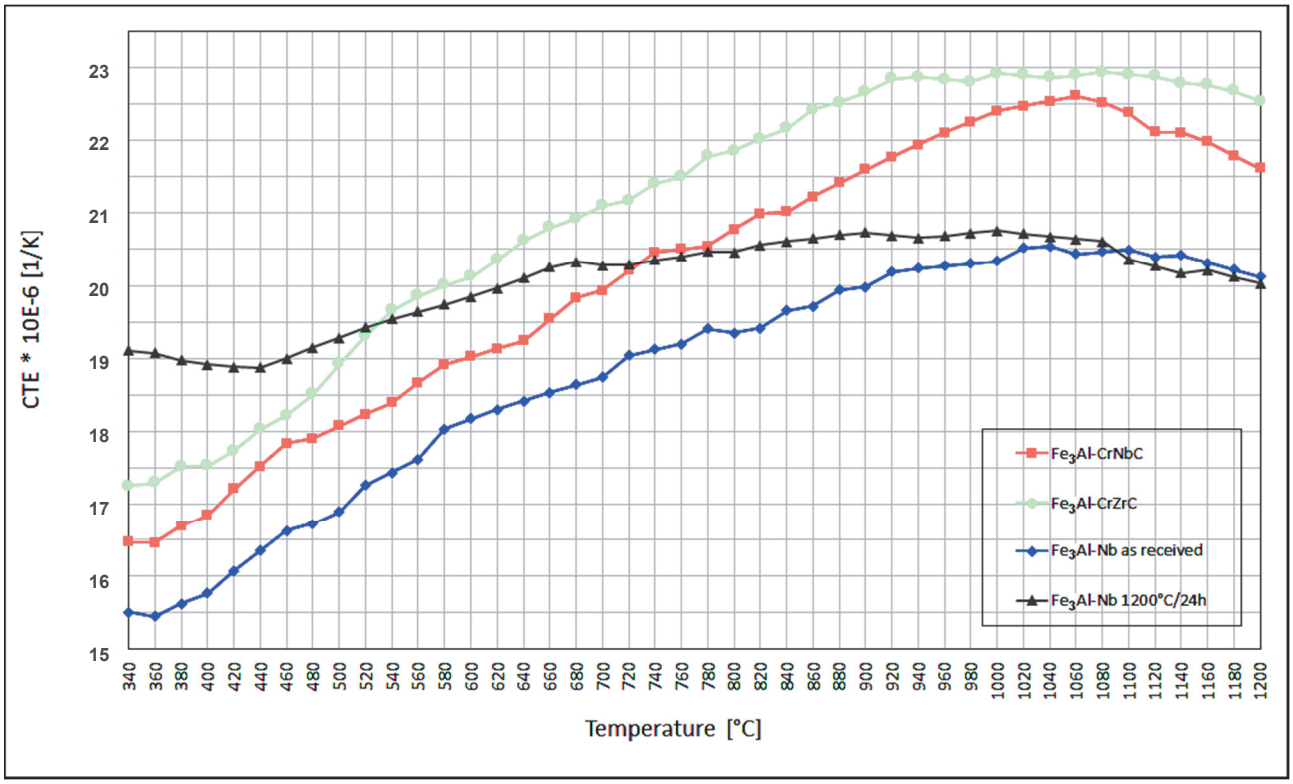


Fig. 6 The chart of the temperature depending of CTE for investigated alloys

CONCLUSIONS

The effect of phase distribution on the values of Coefficient of thermal expansion of Fe_3Al - type iron aluminides was described. The distribution of secondary phase in continual eutectic areas seems to be more advantageous for thermal expansion then presence of individual particles.

The material Fe_3Al-Nb alloyed with the high content of niobium reached the best results of investigated alloys. The reinforcement of the matrix could be caused by "net" of eutectic areas, which could have similar effect as reinforcing phase in composites.

The annealing 24 h at 1200 °C has a significant effect on the CTE values of Fe_3Al-Nb alloy. The CTE values are higher after annealing than in as received state, but they are more stable in a wide temperature range.

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REFERENCES

[1] MC KAMEY, C.G. Iron Aluminides. In *Physical Metalurgy and processing of Intermetallic Compounds.* Eds. STOLOFF N.S., SIKKA V.K., 1994, pp. 351-391.

[2] STOLOFF, N.S. Iron aluminides: present status and future prospects. *Materials Science and Engineering A,* 1998, Vol. *258,* pp. 1-14.

[3] DEEVI, S.C., SIKKA, V.K. Nickel and iron aluminides: an overview on properties, processing and applications. *Intermetallics,* 1996, Vol. *4,* pp. 357-375.

[4] BERDOVSKY, Y.N. - Editor. *Intermetallics Research Progress.* Publ.: Nova Science Publisher, New York 2008. ISBN 978-1-60021-982-5.

[5] PALM, M. Concepts derived from phase diagram studies for the strengthening of Fe - Al-based alloys. *Intermetallics,* 2005, Vol. *13,* pp. 1286-1295.

[6] PALM, M., SCHNEIDER, A., STEIN, F., SAUTHOFF, G. Strengthening of iron aluminide alloys for high-temperature applications. *Mater. Res. Soc. Symp. Proc.,* 2005, *Vol. 842,* S1.7.1-S1.7.12.

[7] MORRIS, D.G., MUNOZ-MORRIS, M.A., REQUEJO, L.M. New iron-aluminum alloy with thermally stable coherent intermetallic nanoprecipitates for enhanced high-temperature creep strenght. *Acta Materialia,* 2006, Vol. *54*, pp. 2335-2341.

[8] MORRIS, D.G. A kol. Strengthening at high temperatures by precipitates in Fe-Al-Nb alloys. *Intermetallics,* 2006**,** *14,* pp. 1204-1207.

[9] ŠVEC, M., HANUS, P., VODIČKOVÁ, V. Coefficient Thermal Expansion of Fe_3Al and FeAl - type iron aluminides. *Manufacturing Technology Journal,* 2013, Vol. 13, No. 3, pp. 399-404.

[10] KRATOCHVIL, P., KEJZLAR, P., KRAL, R., VODICKOVA, V. The effect of Zr addition on the structure and high temperature strength of Fe-30 at.% Al type alloys. *Intermetallics,* 2012, Vol. *20,* pp. 39-46.

[11] PÍŠEK, F. *Nauka o materiálu II.* 2. edition. Československa akademie ved, Publishing: Pratur, 1959.

[12] NETSCH. DIL 402 PC - Horizontal pushrod dilatometer - product brochures. Netsch.com [cit. 2012-10-30]. Available from: http://www.netzsch-thermal-analysis.com/uploads/tx_nxnetzschmedia/files/DIL_E_0313_10.pdf

[13] KANAGARAJ, S., PATTANAYAK, S. Measurement of the thermal expansion of metal and FRPs. *Cryogenic,* 2003, Vol. 43, No. 7, pp. 399-424.

[14] PORTER, W.D., MAZIASZ, P.J. Thermal expansion data on several iron-and nickel-aluminide alloys. *Scripta Metallurgica et Materialia,* 1993, Vol. 29, No. 8, pp. 1043-1048.

[15] RAGHAVAN, V. Al-Cr-Fe (Aluminium-Chromium-Iron). *Journal of Phase Equilibria and Diffusion,* 2012, Vol. 33, Issue 1, pp. 55-58.

[16] ŠVEC, M., VODIČKOVÁ, V., HANUS, P. The effect of heat treatment on the structure of Nb and Cr doped Fe_3Al alloy. In *Metal 2012 conference*. ISBN 978-80-87294-29-1.

[17] EFFENBERG, G., ILYENKO, S. - Editors. *Ternary alloy systems*. Springer Publishing: Berlin 2009. ISBN 978-3-540-88052-3.

[18] RAGHAVAN, V. Al-Fe-Nb (Aluminium-Iron-Niobium). In *Journal of Phase Equilibria and Diffusion,* 2010, Vol. 31, No. 2, pp. 166-167.