

# The Effect of Laves Phase (Fe,Al)<sub>2</sub>Zr on the High-Temperature Strength of Carbon-Alloyed Fe<sub>3</sub>Al Aluminide



PETR KRATOCHVÍL, VĚRA VODIČKOVÁ, ROBERT KRÁL, and MARTIN ŠVEC

The effects of carbon on the phase structure and on the yield stress  $\sigma_{0.2}$  in the temperature range from 873 K to 1073 K (600 °C to 800 °C) of the Fe<sub>3</sub>Al type aluminides alloyed by Zr are analyzed. Four alloys with Zr and C in ranging from 1.0 to 5.0 at. pct of additives were used. The appearing of either Laves phase (Fe,Al)<sub>2</sub>Zr and/or carbides depend on the difference in concentrations,  $c_{Zr} - c_C$ . This parameter ( $c_{Zr} - c_C$ ) has been selected instead of the concentration ratio  $c_{Zr}/c_C$  used in previous works since it exhibits a significantly better correlation with the Laves phase concentration which influences the high-temperature yield stress,  $\sigma_{0.2}$ , of the tested alloys. The presence of Laves phase or eutectic (matrix—Laves phase), respectively, enhances the value of the yield stress  $\sigma_{0.2}$ . The amount of Laves phase is decreased by the presence of C due to the affinity of carbon to Zr.

DOI: 10.1007/s11661-015-3309-2

© The Minerals, Metals & Materials Society and ASM International 2015

## I. INTRODUCTION

THE alloying by zirconium is a method to enhance HT mechanical properties of Fe<sub>3</sub>Al alloys. Very low solid solubility of Zr in the Fe-Al initiated investigations of the influence of Zr addition to Fe<sub>3</sub>Al and FeAl alloys. The beneficial effect of zirconium and niobium addition on *e.g.* creep resistance was first reported in papers by McKamey and Maziasz.<sup>[1–3]</sup> Significant attention was paid to the effect of Zr on the phase structure and high-temperature (HT) mechanical properties of iron aluminides. Especially the formation of Laves phase (Fe, Al)<sub>2</sub>Zr and (Fe, Al)<sub>2</sub>Nb was referred.<sup>[4–10]</sup>

Aside are left the large additions of Zr (*i.e.*, up to 30 at. pct<sup>[4,5]</sup>). It is important to determine the effect of carbon (present in the raw iron used for the preparation of iron aluminides for structural uses). This may modify the mechanical properties, corrosion process, *etc.*, by the influence of, *e.g.*, the Laves phase. The presence of carbon in iron aluminides with zirconium modifies the formation of Laves phase in the Fe corner of the ternary equilibrium diagram.<sup>[6]</sup> Low solubility of Zr in D0<sub>3</sub>/B2 Fe-Al results in the formation of Laves phase (Fe,Al)<sub>2</sub>Zr.

Recently, Kratochvil *et al.*<sup>[11]</sup> showed in iron aluminides alloyed by Cr that the ratio of zirconium and carbon influences the phase structure of Fe<sub>3</sub>Al-type alloys. This was documented by the effects of the ratio

$c_{Zr}/c_C$  on both the yield stress 0.2 and the creep rate at high temperatures.

Laves phase  $\lambda_1$  (Fe,Al)<sub>2</sub>Zr and ZrC particles were found primarily along grain boundaries, with some distribution in the grains as well. Their strengthening role is limited to blocking the grain boundaries' motion because the effect of these particles inside the grains (owing to large distance between the particles) was very small. Partly such effects of low concentrations of Zr and C have been mentioned by Kejzlar *et al.*<sup>[12]</sup> The appearing of metastable phases, (Fe<sub>1-x</sub>Al<sub>x</sub>)<sub>3</sub>Zr and Fe<sub>2</sub>Zr, which originate during the HT deformation<sup>[10]</sup> was also detected in the iron aluminides alloyed by Cr.

It is therefore the purpose of the present paper to verify the effect of the ratio of zirconium and carbon—described by the difference  $c_{Zr} - c_C$ —in an alloy without chromium on the structures of Fe<sub>3</sub>Al alloys and on their HT yield strengths. The difference  $c_{Zr} - c_C$  was chosen as it better describes the amount of Zr left for the formation of Laves phase, if any, compared with the ratio of both concentrations. This fact is obvious from a significantly better correlation with eutectic (matrix—Laves phase) in case of  $c_{Zr} - c_C$  compared with  $c_{Zr}/c_C$ .

## II. EXPERIMENTAL PROCEDURE

The alloys were prepared by a vacuum melting and casting under argon. The chemical compositions are given in Table I. The actual compositions of the alloys were established by wet chemical analysis at the Research and Testing Centre, Plzen. The concentrations were chosen to get alloys with different values of  $c_{Zr} - c_C$ . The alloys contain (25.6 to 26.2) at. pct Al. Concentrations of impurities (from the metals used for

PETR KRATOCHVÍL, Professor, and ROBERT KRÁL, Associate Professor, are with the Department of Physics of Materials, Charles University, Ke Karlovu 5, 121 16 Prague, Czech Republic. Contact e-mail: pekrat@met.mff.cuni.cz VĚRA VODIČKOVÁ, Associate Professor, and MARTIN ŠVEC, Ph.D. Student, are with the Department of Materials Science, Faculty of Engineering, Technical University of Liberec, Studentská 2, 46117 Liberec, Czech Republic.

Manuscript submitted September 10, 2015.

Article published online December 30, 2015

the preparation of the alloys) were: 0.1 at. pct Cr, 0.01 at. pct B, 0.1 at. pct Mn.

All alloys were tested in the as-cast state. Samples (parallelepipeds  $6 \times 6 \times 10$  mm) for compression test at temperatures ranging from 873 K to 1073 K (600 °C to 800 °C) were prepared from the alloys via electrical discharge machining (EDM). The compressive yield stress was evaluated using a digitally controlled testing machine (INSTRON 1186R). The deformation rate was  $1.2 \times 10^{-4} \text{ s}^{-1}$ .

The temperatures ranging from 873 K to 1073 K (600 °C to 800 °C) were chosen because these temperatures are mostly above the yield strength anomaly. This is an increase in the strength with the increasing temperature, typically observed for Fe-Al-based alloys with a maximum at about 823 K (550 °C). All the investigated alloys are at these temperatures, B2-ordered, which allows direct comparison of their yield strengths.

Microstructural phase compositions were studied using a field emission-scanning electron microscope (FE-SEM)—Zeiss Ultra Plus—equipped with energy-dispersive spectrometer (EDS)—OXFORD X-MAX 20—and with a detector for electron backscatter diffraction (EBSD)—OXFORD NordlysNano. For the characterization of the carbides, EDS combined with analysis of Kikuchi patterns was used. Analyses were carried out at an accelerating voltage of 20 kV, and the typical primary electron beam diameter was 2 nm.

### III. EXPERIMENTAL RESULTS AND DISCUSSION

The sizes of the grains of all the investigated as-cast alloys were ranging from 100 to 400  $\mu\text{m}$ . The phase structures of the alloys are summarized in Table I. In alloy 1143 ( $c_{\text{Zr}} - c_{\text{C}} = -0.67$  at. pct), the volume fraction  $f_v$  of ZrC is 1.9 pct. ZrC are plates partly seen edgewise. The rest of the carbon forms perovskite  $\text{AlFe}_3\text{C}_{0.5}$  ( $f_v = 4.5$  pct) as documented in Figure 1 or is dissolved in the lattice. Perovskite is forming also plates, contrast of which is not excellent. In alloy 1144 ( $c_{\text{Zr}} - c_{\text{C}} = 0.53$  at. pct), ZrC particles ( $f_v = 2.3$  pct) and Laves phase  $(\text{Fe,Al})_2\text{Zr}$  ( $f_v = 4.2$  pct) appear. The Laves phase is a part of an eutectic with matrix, Figure 2.

The structures of alloys 1181 and 1182 are characterized by a dendritic arrangement of eutectic (matrix plus Laves phase) immersed in the matrix, Figures 3 and 4. ZrC carbide particles are seen inhomogeneously dispersed, and its volume fraction  $f_v = 1.2$  pct in 1181 alloy. In 1182 alloy,  $f_v$  reaches the value of 3.9 pct. The values of  $f_v$  of the eutectic Laves phase plus matrix are 41.7 and 34.7 pct, respectively, in the both the alloys. The basic

point of the above observations is that the eutectic strengthens the tested alloys.

The compression 0.2 yield stresses as a function of temperature was used to describe HT mechanical properties of the tested iron aluminides, which are summarized in Table II. Experimental errors are 3 pct (valid also for yield stress 0.2 values in Table II). The increasing value of HT yield stress 0.2 with the increasing volume fraction of the eutectic (Laves phase  $\lambda_1$   $(\text{Fe,Al})_2\text{Zr}$  with matrix) is obvious as seen in Figure 5. Due to the fact that carbon forms ZrC, it lowers the amount of Zr available for the formation of Laves phase  $\lambda_1$   $(\text{Fe,Al})_2\text{Zr}$ . Figure 5 shows the comparison of the

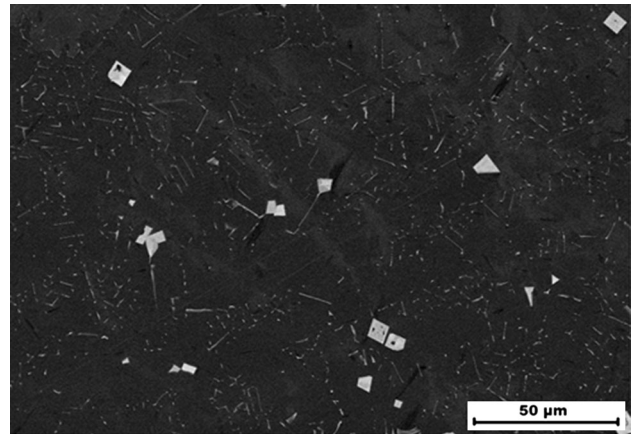


Fig. 1—The structure of the as-cast 1143 alloy (SEM): perovskite black (dark needle like), carbide—white, matrix—gray.

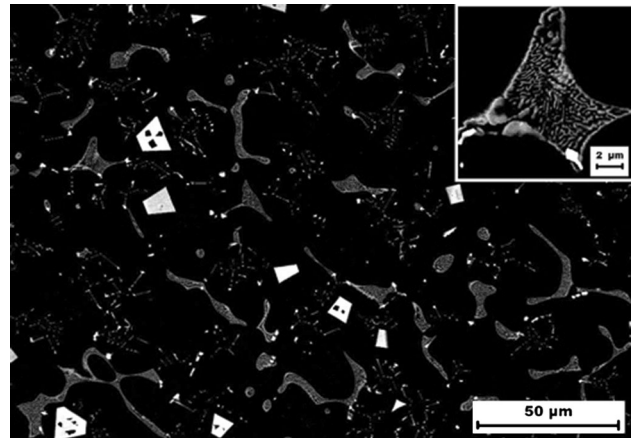


Fig. 2—The structure of the as-cast 1144 alloy (SEM): carbide—white (polygonal, partly rod like), multiform eutectic of Laves phase and matrix—gray, matrix—dark.

Table I. The Compositions of Alloys

	Zr	C	$c_{\text{Zr}} - c_{\text{C}}$ (at. pct)	$f_v$ (pct) ZrC	$f_v$ (pct) Eutectic
1143	1.05	1.72	-0.67	1.9 + 4.5 (perovskite)	—
1144	2.11	1.58	0.53	2.3	4.2
1181	4.9	1.4	3.5	1.2	41.7
1182	4.8	2.1	2.6	3.9	34.9

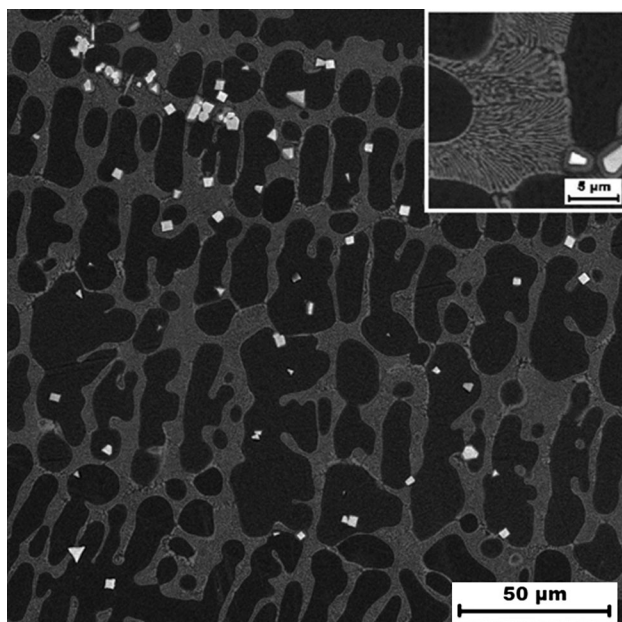


Fig. 3—The phases in the as-cast alloy 1181 (SEM): eutectic of Laves phase, and matrix—gray, ZrC carbide—white, matrix—dark, with the details shown in the upper right corner.

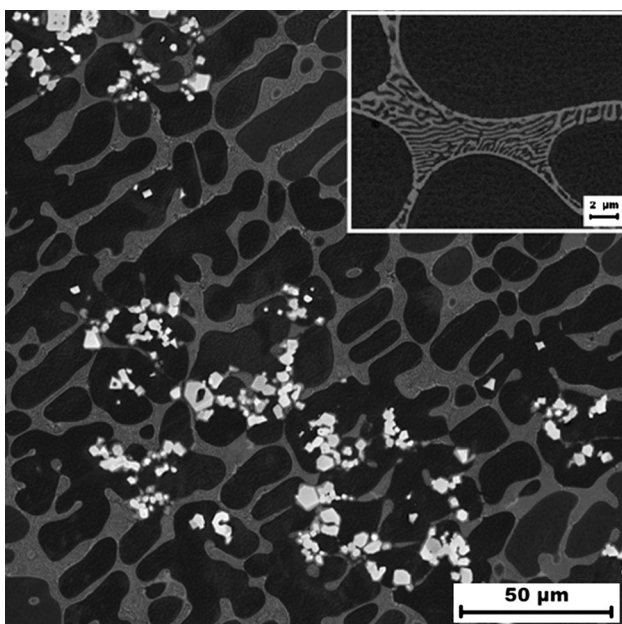


Fig. 4—The phases in the as-cast alloy 1182 (SEM): eutectic of Laves phase and matrix seen as gray, ZrC carbide as white, matrix as dark, the detail in the upper right corner.

curves (1144, 1181, and 1182) with  $c_{Zr} - c_C > 0$  with the curve of alloy 1143 with  $c_{Zr} - c_C < 0$ . This relationship (the influence of concentration of zirconium and carbon) between the four alloys studied is kept even during further deformation, *i.e.*, up to the maximum stress (given also for all alloys and temperatures in Table II). Maximum stress is reached between 5 and 10 pct. compressive strain. The lengths of error bars are comparable with the sizes of symbols, and the bars in Figure 5 are not plotted. The effect is less pronounced at 1073 K (800 °C) probably due to the softness of the matrix. The main effect of softening by the exhaustion of Zr in the aluminide by formation of the ZrC remains.

The solubility of Nb in iron aluminide  $Fe_3Al$  is low compared with Zr. For the similarity of ternary diagrams in respect of Fe-Al-Zr and Fe-Al-Nb with Laves phase in Fe corners, see References 13 and 14. The phase structure and the HT strength of the Fe-26Al-4Nb-1C and Fe-26Al-2Nb-1C alloys are compared in References 15 and 16. The structures of both alloys include NbC carbide and  $(FeAl)_2Nb$  Laves phase. Only nominal values of  $c_{Nb} - c_C$  for the mentioned alloys are available. Thus, it correlates to the above reasoning that the presence of the Laves phase is reported only in the Fe-26Al-4Nb-1C alloy, for which higher values of yield stress 0.2 at HT were observed.

#### IV. CONCLUSIONS

The amount of the Laves phase  $\lambda_1 (Fe,Al)_2 Zr$  in  $Fe_3Al$  aluminides is influenced by the presence of carbon (great

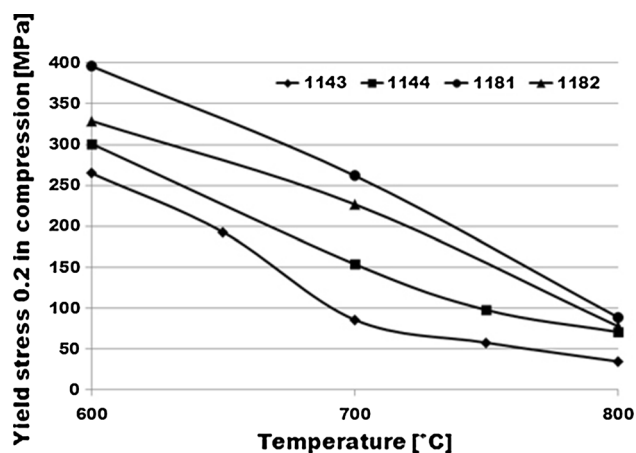


Fig. 5—The dependence of the yield stress 0.2 on temperatures ranging from 873 K to 1073 K (600 °C to 800 °C) for as-cast alloys 1143, 1144, 1181, and 1182.

Table II. Yield Stress 0.2/Maximum Stress of the Tested Alloys (in MPa)

Temperature [K (°C)]	1143	1144	1181	1182
873 (600)	265/408	301/453	396/576	329/518
923 (650)	193/239	—	—	—
973 (700)	86/151	154/202	262/348	227/309
1023 (750)	58/90	98/135	—	—
1073 (800)	35/58	71/82	89/118	78/111



affinity of C to Zr). The formation of ZrC carbides has a negative effect on the HT strength of Fe<sub>3</sub>Al type aluminides, while it lowers the amount of Zr available for the formation of the Laves phase, strengthening the aluminide. The eutectic of Laves phase plus matrix enhances the yield stress 0.2 at high temperatures.

The behaviors of Nb- and C-alloyed iron aluminides reported elsewhere can be understood in the same way. For the use Zr or Nb as an additive to iron aluminides in structural materials, the presence of even small concentrations of carbon must be taken in account.

#### ACKNOWLEDGMENT

The paper is based on the work supported by the Grant Agency of the Czech Republic within the Project 108/12/1452.

#### REFERENCES

1. C.G. McKamey, P.J. Maziasz, and J.W. Jones: *J. Mater. Res.*, 1992, vol. 7, pp. 2089–2106.

2. C.G. McKamey, P.J. Maziasz, G.M. Goodwin, and T. Zacharia: *Mater. Sci. Eng. A*, 1994, vol. 174, pp. 59–70.
3. C.G. McKamey and P.J. Maziasz: *Intermetallics*, 1998, vol. 6, pp. 303–14.
4. A. Wasilkowska, M. Bartsch, F. Stein, M. Palm, K. Sztwiertnia, G. Sauthoff, and U. Messerschmidt: *Mater. Sci. Eng. A*, 2004, vol. 380, pp. 9–19.
5. A. Wasilkowska, M. Bartsch, F. Stein, M. Palm, G. Sauthoff, and U. Messerschmidt: *Mater. Sci. Eng. A*, 2004, vol. 381, pp. 1–15.
6. F. Stein, M. Palm, and G. Sauthoff: *Intermetallics*, 2005, vol. 13, pp. 1275–85.
7. D.G. Morris, M.A. Muñoz-Morris, and L.M. Requejo: *Acta Mater.*, 2006, vol. 54, pp. 2335–41.
8. D.G. Morris, I. Gutierrez-Urrutia, and M.A. Muñoz-Morris: *Scripta Mater.*, 2007, vol. 57, pp. 449–52.
9. D.G. Morris and M.A. Muñoz-Morris: *Mater. Sci. Eng. A*, 2012, vol. 552, pp. 134–44.
10. D.G. Morris, L.M. Requejo, and M.A. Muñoz-Morris: *Intermetallics*, 2012, vol. 13, pp. 862–71.
11. P. Kratochvíl, F. Dobeš, J. Pešíčka, P. Málek, J. Buršík, V. Vodičková, and P. Hanus: *Mater. Sci. Eng. A*, 2012, vol. 548, pp. 175–82.
12. P. Kejzlar, P. Kratochvíl, R. Král, and V. Vodičková: *Metall. Mater. Trans. A*, 2014, vol. 45A, pp. 335–43.
13. F. Stein, G. Sauthoff, and M. Palm: *Z. Metallk.*, 2004, vol. 95, pp. 469–76.
14. M. Palm: *J. Alloy Compd.*, 2009, vol. 475, pp. 173–77.
15. A. Schneider, L. Falat, G. Sauthoff, and G. Frommeyer: *Intermetallics*, 2003, vol. 11, pp. 443–50.
16. L. Falat, A. Schneider, G. Sauthoff, and G. Frommeyer: *Intermetallics*, 2003, vol. 13, pp. 1256–62.