ISSN 2229-5518

Microstructural Stability of Spray-atomized FeAl Alloys

M. Amaya, J.M. Romero, L. Martínez and R. Perez

Abstract— The role of boron and alumina particulate on the microstructural stability of as-atomized FeAl40at% base alloys in the temperature range of 600-1200°C was evaluated. The alumina particulate reinforcement combined with the boron microalloying prevents the abnormal grain growth and enhances the microstructural stability. The anomalous grain growth behavior which occurred in the temperature range of 600-900 °C was related with an onset of the recrystallization. The presence of a hardness peak observed at 900°C in the as-atomized FeAl base alloy was strongly diminished with the boron microalloying and alumina particulate reinforcement.

Index Terms— iron aluminides, abnormal grain growth, annealing, microalloying

1 Introduction

THE FeAl intermetallic alloys have been attractive for their superior performance in mechanical properties and corrosion resistance at high temperatures [1-4]. However, their low ductility at room temperature has limited many practical applications [5-6]. Spray-atomization and deposition metallurgical technique improves the microstructural and mechanical properties in the FeAl intermetallic alloys [7]. Also, this technique facilitates the incorporation of strengthening second phases that improves creep and other mechanical properties [8]. The intrinsic lack of ductility in FeAl intermetallic alloys has been limiting the manufacturing of useful commercial products. For this reason, the possibility of application of iron aluminides as surface coatings may be considered to take advantage of their superior corrosion performance at high temperatures [4]. Recently, the oxidation behavior of low-pressure plasma sprayed iron aluminides were explored [9]. Several studies of grain growth [10-12] and recrystallization [14-19] have also been reported. These studies include dynamic recrystallization, abnormal grain growth in textured alloys, boron effect on the recrystallization, and anomalous softening prior to static recrystallization. In the present study the grain growth kinetics of spray-atomized FeAl40at% alloys after annealing experiments were evaluated.

2 EXPERIMENTAL PROCEDURE

Ingots of FeAl40at.% (FA), FeAl40+0.1at.%B (FAB), and FeAl40+0.1at.%B+10at.%Al2O3 (FAL) spray atomized intermetallic alloys were used to fabricate cubic shape specimens with dimensions of 5x5x5 mm. A spark-cutting machine was employed to cut 15 specimens from each ingot. Details of fabrication of the ingots were discussed elsewhere [7]. All specimens were mechanical grinded on wet SiC-paper (grit 240-600).

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Finally, the specimens were polished using colloidal alumina of 1 □m. Before the annealing studies, the grain structure was revealed using a solution consisted in 33% nitric acid, 33% acetic acid, 33% H2O and 1% flourhidric acid (% volume). The intercept method (ASTM-E112) was employed to measure the grain sizes before and after of the annealing experiments. More than 300 grains were counted for each specimen. Microhardness measurements were performed with a 200 g load and 20s of holding time. Annealing experiments at various temperatures for various periods were performed in an automatic-control electric furnace (times: 1, 12, 24, 48 and 72 h, temperatures: 600, 900 and 1200°C). After the annealing processes, the power supply was switched off and the furnace was cooled down to room temperature at its natural rate. Finally, the specimens were polished and etching, and then the grain sizes were measured using the procedure described above. This is illustrated in figure 1. In order to visualize the porosity and alumina particulate distribution in the microstructure, the specimens were observed by optical microscopy.

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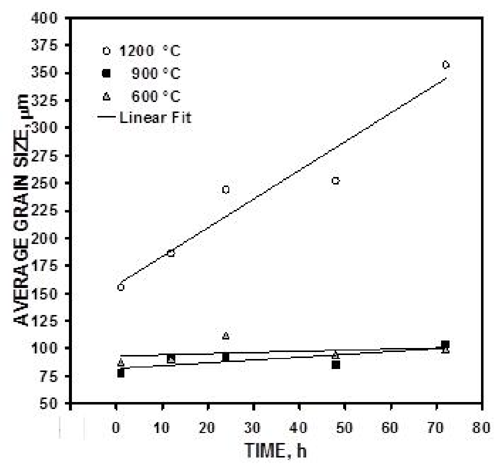

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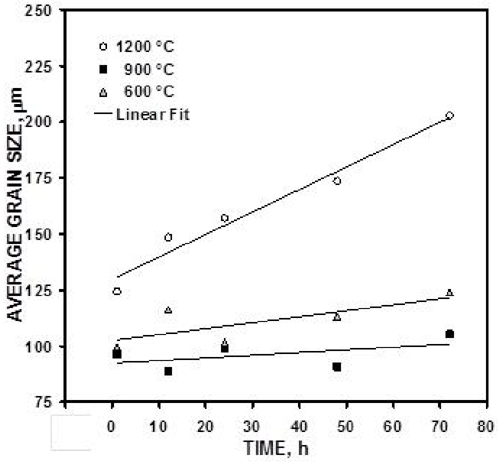

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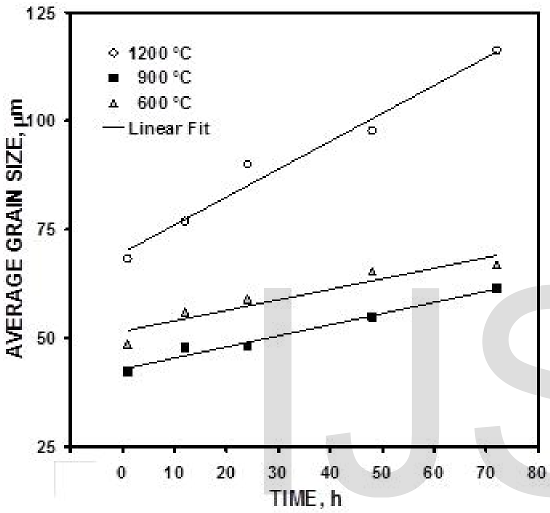

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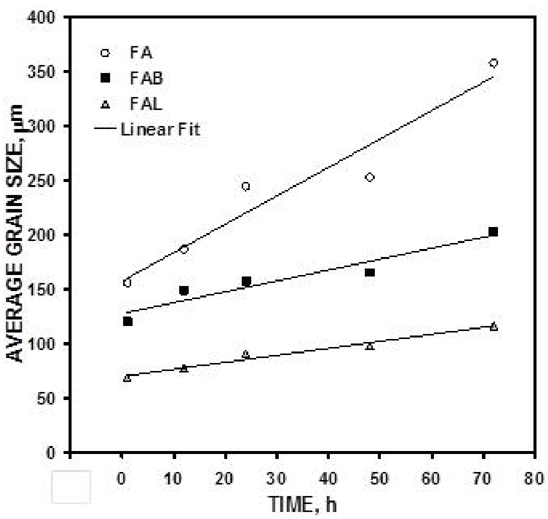


(d

Figure 1. Average grain size as a function of annealing time for the (a) FA, (b) FAB, (c) FAL alloys, and (d) effects of boron and alumina particulate on the grain growth kinetic at 1200°C in the three iron aluminides.

3 RESULTS AND DISCUSSION

3.1 GRAIN GROWTH KINETICS

The grain growth kinetics of the FA alloy is illustrated in figure 1a. The grain growth kinetic was not described by any normal grain growth fit law equation in all temperature range. The non-uniform initial grain size distribution could induce this last behavior, but a reasonably linear fit describes the grain growth of the FA alloy at 1200°C, (see Figure 1a). The results also indicated that in the temperature range of 600-900 °C, the grain growth kinetics were practically the same.

The grain growth kinetics of the FAB alloy is illustrated in Figure 1b. Basically, the fit of the grain growth data of the FAB alloy follows a similar behavior of the FA alloy. However, the grain growth kinetics of the FAB alloy diminishes considerably at 1200°C as compared with the FA alloy. At this temperature a good linear fit of the grain growth data with a statistical coefficient of determination (R2) of 0.9668 was obtained. The final grain size distribution at 1200 °C was more uniform compared as the FA alloy. In this case is evident that the boron microalloying plays a key role in the diminished grain growth rate at 1200°C. The boron effect on the grain growth of iron aluminides was observed previously for Yang and Baker [14]. They proposed that the boron atoms presumably form boron atomic environments around dislocations, reducing their mobility, as well as reducing also the grain boundary mobility. The boron segregation influences the structure of lattice defects and segregation also could be occurring in planar or line defects in the FeAl alloys [20]. The results also indicated that in the temperature range of 600-900 °C anomalous grain growth kinetics was presented. In this case the average grain growth kinetics at 900 °C was lower compared to the average grain growth kinetics at 600 °C. Recently, Bystrzycki and Varin [15] founded a recrystallization onset in specimens of FeAl40at.% doped with Zr and B deformed by shock-waves, after annealing experiments during 1 hour at 700°C, and a completely recrystallized microstructure at 800°C. Also, Yang and Baker observed similar behavior in FeAl40at.% doped with boron [14]. In the present study, the anomalous grain growth kinetics observed in the temperature range of 600-900 °C suggest that the presence of a recrystallization onset occurred in the last temperature range. The fabrication procedure suggests [7] the presence of the residual stresses generated by the high-impacts and thermal shock resulted during the deposition of the FeAl spray. The driving force that induced this last recrystallization onset could be generated from these residual stresses. It is known that in normal grain growth the average grain size increases without essential changes in the size and shape distributions [21]. During normal grain growth the logarithm of the grain diameter, the number of neighbors (or sides) in a planar intersection, and

ISSN 2229-5518

the number of faces in three dimensions can all be approximated by Gaussian normal distributions [22-23]. Also, normal grain growth is controlled by the rate of loss of grains from the lowest topological class. The topological distribution after 24 hours of annealing in the FAB alloy is shown in the Figure 2. It be can shown that at 900 °C the frequency of grains with topological classes of 3, 4, 5 and 6 were larger as compared to the frequency of grains with topological classes above 7. This typical distribution reflects the recrystallization onset occurred at this temperature. Also, the most stable topological class distribution observed at 1200 °C confirms the grain growth kinetics behavior occurred at this temperature.

The grain growth kinetics of the FAL alloy is shown in figure 1c. A reasonably linear fit describes the grain growth of the FAL alloy in all temperature range, but only at temperatures of 900 °C and 1200 °C good R2 values of 0.9755 and 0.9739 respectively were obtained. In the same way that the FAB alloy, the FAL alloy showed anomalous grain growth kinetics in the temperature range of 600-900 °C. In the same way that in the FAB alloy, this last behavior could

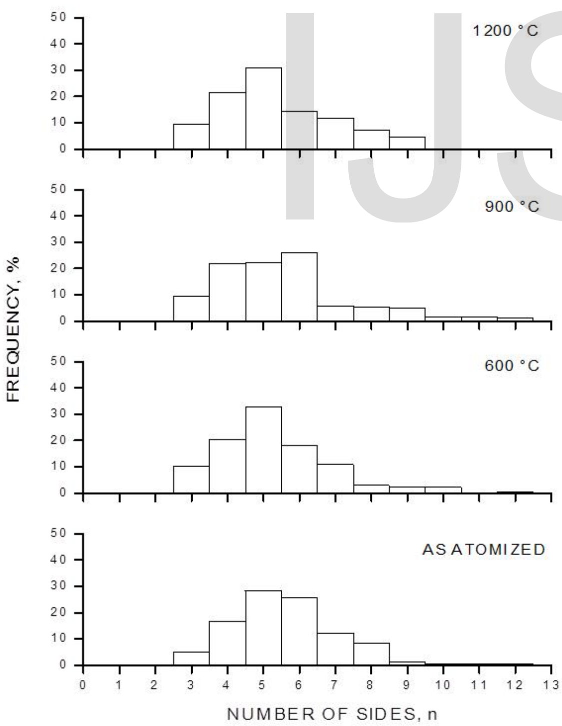
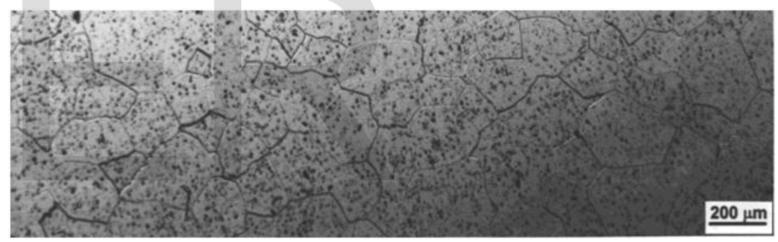


Figure 2. Number of sides per grain distribution for the FAB alloy annealed during 24 h.

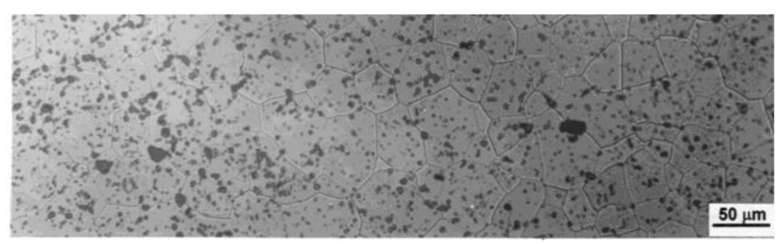
occur by an onset of recrystallization. Compared withs the FAB alloy, the gap between the linear fit in the temperature range of 600-900 °C were about 2 times smaller than the FAL alloy. This last result suggests that the reinforcement with alumina particulate diminish the effects of the boron microalloying on the grain boundaries, by the grain size reduction. Consequently, the recrystallization mechanism could be affected.

3.2 MICROSTRUCTURE CHARACTERISTICS

In the Figure 3, the as-atomized microstructures of the three iron aluminides alloys before the annealing experiments are presented. The FA and FAB alloys (Figure 3a and 3b, respectively) showed noticeably porosity as compared to FAL alloy (Figure 3c). In this case, the reinforcement with alumina particulate (Al2O3) prevents the porosity formation in the microstructure (see detail of the Figure 4). Not noticeable effect on the grain size refinement was induced by the boron microalloying. However the presence of Al2O3 alumina particulate induced a grain size refinement and improves the microstructure of the FAL alloy. The initial mean grain sizes for the FA, FAB, and FAL intermetallic alloys were 77, 79 and 33 □m, respectively. After the annealing experiments the average grain sizes as a function of the annealing time (t) were plotted. The grain growth curves for the FA, FAB, and FAL alloys were showed in the figures 1a, 1b, and 1c, respectively.



(a)



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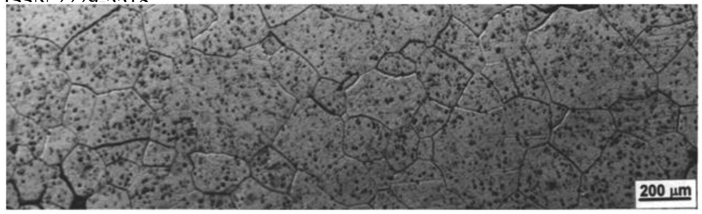


(c)

Figure 3. As-atomized microstructures of the (a) FA, (b) FAB, and (c) FAL iron aluminides

3.3 MICROSTRUCTURAL STABILITY

In the present study the microstructural stability was discussed after that the recrystallization onset was occurred. The figure 1d shows a plot of grain growth kinetics for the three FeAl alloys tested at 1200 °C. The results indicated that the grain growth stability at 1200°C was most notable in the FAL alloy, less notable in the FAB alloy, and least notable in the FA alloy. Since the initial average grain size in the FAL alloy was about 2 times smaller than the FAB alloy, it was conceivable a possible reduction also of about 2 times of boron concentration along the grain boundary. This suggests that the boron microalloying plays a key role in the last behavior. Also, the results indicated that the alumina particulate stabilize the grain growth kinetics and prevents the abnormal grain growth. The microstructural evolution after the annealing

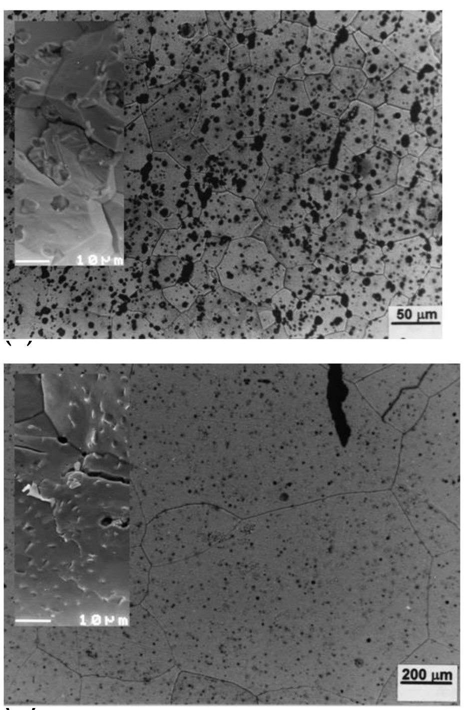


Figure 4. (a) Normal grain growth in the FAL alloy and (b) abnormal grain growth in the FA alloy annealed at 1200 °C for 48 h.

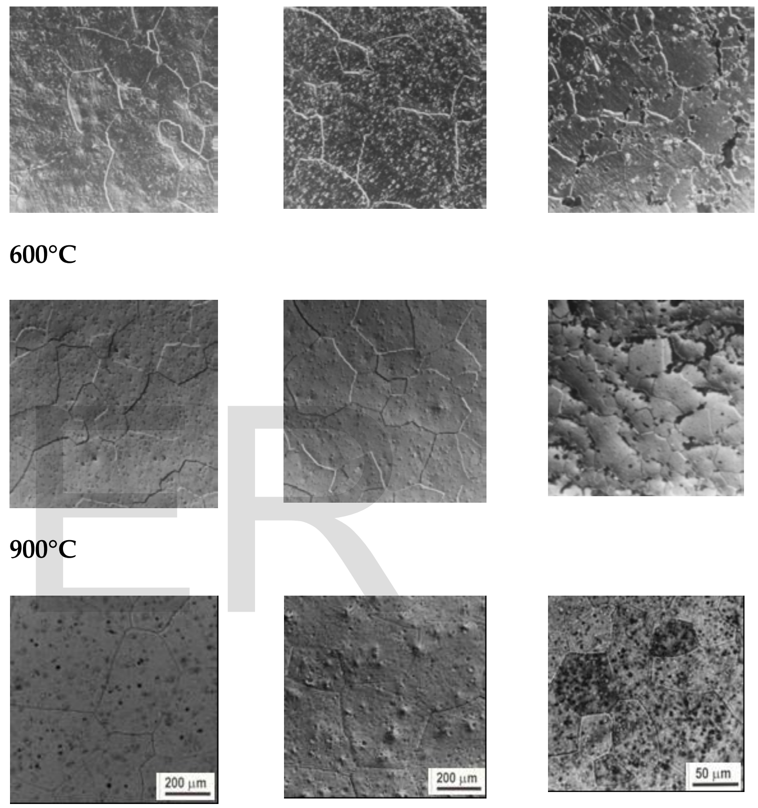
experiments for 12 hours is shown in the Figure 5. It can be seen the better grain growth stability in the FAL alloy compared with the FAB and FA alloys. Also the anomalous grain growth at 900 °C was clearly shown. The microhardness as a function of annealing temperature at various times is presented in the Figure 6. The FA alloy showed a hardness peak above 900 °C at all the evaluated annealing times (see Figure 6a). In the most ordered

FA **FAB** **FAL**

600°C

900°C

1200°C



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intermetallics alloys, the hardness depends both on the change of dislocation density and on the change of long-range order degree. The hardness behavior in the FA alloy suggests that a reduction in dislocation density decreases

Figure 5. Microstructural evolution after 12 hours of annealing of the three iron aluminides

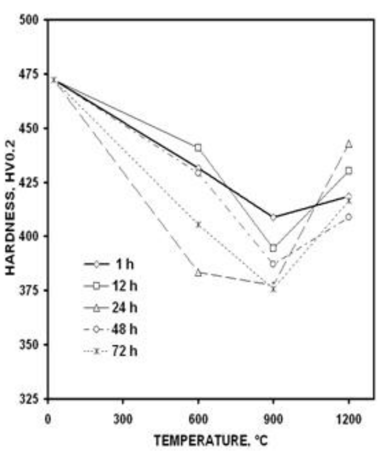
.the hardness from room temperature to 900 °C. The results also suggest that the increases in the hardness above 900 °C could be promoted by a strong reordering in the long-range order degree during the annealing. In the case of the FAB and FAL alloys, both alloys showed a reduction of hardness at all evaluated times and temperatures. In this case, the addition of boron and alumina particulate affects the dependence of hardness on reordering above 900 °C, as

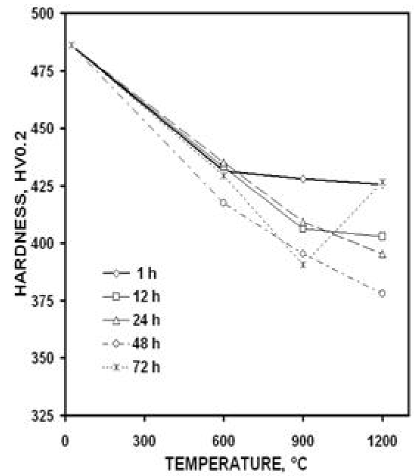
ISSN 2229-5518

compared with the behavior observed in the FA alloy. One exception occurred in the hardness behavior after 72 h of annealing in both FAB and FAL alloys. In the same way that in the FA alloy, a hardness peak above 900 °C was presented. This last result suggests that at long annealing times, the effect of boron and alumina particulate on the reordering that affect the hardness, was eliminated.

4 CONCLUSIONS

The grain growth data in the all three FeAl intermetallic alloys was not described by any normal grain growth fit law equation. Only FAL and FAB alloys shows a good linear fit at 1200 °C and at 900 °C, respectively. Anomalous grain growth kinetics was observed in the temperature range of 600-900 °C in both FAB and FAL alloys. The possible presence of residual stresses generated by the high-impacts and thermal shock resulted during the deposited of the alloys suggest the occurrence of a recrystallization onset in the last temperature range. The results indicated that the grain growth stability at 1200°C was most pronounced in the FAL alloy, less pronounced in the FAB alloy, and more less pronounced in the FA alloy. The alumina particulate combined with boron microalloying stabilizes the grain growth kinetics and prevents the abnormal grain growth. The FA alloy showed a hardness peak above 900 °C at all evaluated annealing times. This result suggests that the increases in the hardness above 900 °C could be promoted by a strong reordering in the long-range order degree during the annealing. The FAB and FAL alloys, showed hardness reduction at all evaluated times and temperatures. One exception occurred in the hardness behavior after 72 h of annealing, where a hardness peak above 900 °C was presented. The addition of boron and alumina particulate affects the dependence of hardness above of 900 °C, but at long times of annealing (above 72 h) this effect was eliminated.





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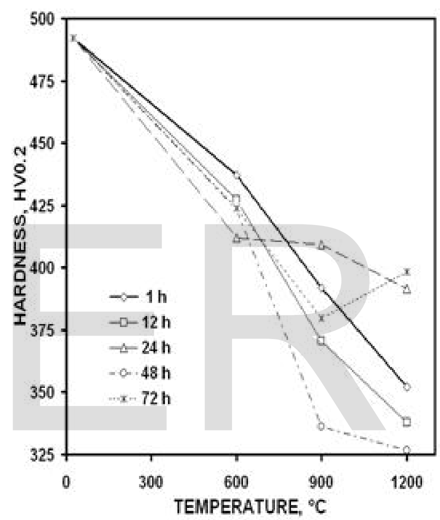

(c)

Figure 6. Microhardness as a function of the annealing temperature, (a) FA, (b) FAB, and (c) FAL alloys.

ACKNOWLEDGEMENTS

The authors acknowledge the valuable technical support of J. C. Arillo Campos and Osvaldo Flores.

REFERENCES

[1] Pike, L. M. & Liu, C. T., Scripta Metall. et Mater, 1991, 25, p. 2757.
[2] Baker, I. & Nagpal, P., in Structural Intermetallics, ed. R. Darolia et al. TMS, Warrendale, USA, 1993, p. 463.
[3] Sikka, Vinod, K., in Processing, Properties, and Applications of Iron Aluminides, ed. J. H. Scheibel & et al. TMS, Warrendale, USA, 1994, p. 3.
[4] M. Amaya, J. Porcayo-Calderon, and L. Martinez, Corrosion, 57, 2001, 489.
[5] Fu, C. L. & Yoo M. H., Acta metall. Mater, 1992, 40, p. 703.
[6] Webb, G., Juliet, P. & Lefort, A., Scripta Metall. et Mater, 1993, 28, p. 769.
[7] Martinez, L., Flores, O., Amaya, M., Duncan, A., Viswanathan, S., Lawrinowics, D. & Lavernia, E. J., Journal of Materials Synthesis and Processing, 5, 1997, p. 65.
[8] Martinez, L., Amaya, M., Flores, O, Lawrinowics, D. & Lavernia, E., in Creep

ISSN 2229-5518
and Fracture of Engineering Materials and Structures, ed. J. C. Eartman et al. TMS, Warrendale, USA, 1997, p. 395.¨

[9] Naoya Masahashi, Sadao Watanabe and Shuji Hanada, ISIJ International, 2001, 41, p.1010.

[10] Schmidt, B., Nagpal, P. & Baker, I., in High-Temperature Ordered Intermetallic Alloys III. MRS Symp. Proc., 133, 1989, p. 755.

[11] Stout, J. J. & Crimp. M. A., Mater. Sci. Eng., 1992, A152, p. 335.

[12] Fashing, A. A., Edwards, G. R. & David, S. A., Scripta Metall. et Mater., 1994, 30, p. 1003.

[13] Baker, I. & Gaydosh, D. J., Metallography, 1987, 20, p. 347.

[14] Yang, Y. & Baker I., Scripta Materialia, 1996, 34, p. 803.

[15] Bystrzycki, J. & Varin, R. A., Scripta Materialia, 1998, 38, p. 465.

[16] Froyen, Ludo; Huang, Y. D., Intermetallics, 2002, 10, p. 473.

[17] Sakata T., Coma H., Yasuda H. and Umakoshi Y., ISIJ International, 2002, 42, p.1010.

[18] Skrotzki W., Kegler K., Tamm R. and Oertel C.-G., Materials Science Forum, 2004, 467-470, p. 525.

[19] Guessasma S., Suzon E., Rouag N. and Grosdidier T., Materials Science Forum, 2004, 467-470, p. 975.

[20] Cadel E., Lemarchand D., Gay A.S., Fraczkiewicz A. and Blavette D., Scripta Materialia, 1999, 41 (4), p. 421.

[21] Hsun, Hu. Rath, B. B., Vandermeer, R. A., in Recrystallization ´90, ed. T. Chandra. TMS, Warrendale, USA, 1990, p. 3.

[22] Kurtz, S. K., Carpay, F. M. A., J. Appl. Phys., 1980, 51(11), p. 5727.

[23] Kurtz, S. K., Kumar, S., Banavar, J. R., Zhang, H., in Modeling of Coarsening and Grain Growth, ed. C. S. Pande and S. P. Marsh. TMS, USA, 1993, p. 165.