Grain Boundary Pest of Boron-Doped Ni_3Al at 1200 °C

T.H. CHUANG, Y.C. PAN, and S.E. HSU

A ductile $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy suffered severe intergranular embrittlement after air exposure at 1200 °C for 100 hours. However, the material survived after 1038 °C air exposure for 100 hours. Auger analysis showed enormous oxygen segregation on the grain boundaries in the 1200 °C, air exposed, boron-doped Ni_3Al, while the boron segregation remained unchanged. To elucidate this type of grain boundary damage, a modified fracture mechanism was proposed. Finally, anomalous grain growth was found in this alloy after 1200 °C air exposure, and an explanation for this phenomenon was suggested.

I. INTRODUCTION

"GRAIN boundary pest" is a term for the high-temperature grain boundary embrittlement of intermetallic compounds dubbed during the 1960s. These intermetallic compounds, which are ordinarily oxidation resistant, suffer severe intergranular failure after prior exposure to an oxygen-bearing atmosphere at elevated temperatures. The mechanism of embrittlement has been debated for a long time. Seybolt and Westbrook[1] suggested that "grain boundary pest" was caused by oxygen-induced grain boundary hardening due to oxygen segregation. Another mechanism proposed by Turner *et al.* suggests that "grain boundary pest" in NiAl may be attributed to progressive oxygen penetration down a grain boundary and oxidation of Al_2O to α-Al_2O_3 at grain boundaries.[2] In this case, the disintegration of material could then result from the internal strains produced by the volume expansion involved in the transformation. Such a process would open up the material along the grain boundaries and allow oxygen to diffuse further into the interior in such a way as to continue the transformation.[3] However, α-Al_2O_3 particles precipitated on grain boundaries were not observed by Seybolt and Westbrook.[4] Turner *et al.*[2] also indicated that "grain boundary pest" only occurred at about 1400 °C. However, a critical temperature for the occurrence of the problem seems to be superfluous according to the mechanism of Westbrook *et al.*

The "grain boundary pest" problem debated during the 1960s concerned mainly unstressed intermetallic compounds. In other words, the intergranular embrittlement was observed following a prior air exposure at elevated temperatures. However, a dramatic drop in the ductility for stressed Ni_3Al-based intermetallic compounds has also been reported in many recent studies. Liu and White[5] conducted tensile tests of Ni_3Al in a 10^{-3} Pa vacuum and found that the ductility of the material remained above 40 pct for temperatures up to 600 °C, while it dropped drastically to 5 to 10 pct in the temperature range between 800 °C and 1000 °C. The dramatic loss of ductility was accompanied by a transition from transgranular to intergranular failure. In subsequent works, Liu and coworkers[6,7] tensile tested Ni_3Al-based intermetallic alloys at 600 °C and demonstrated that a change in test environment from vacuum to air caused a dramatic drop in ductility from about 50 pct to 1 to 7 pct. Taub *et al.*[8] similarly discovered the severe intergranular embrittlement in as tensile-tested Ni_3Al above 400 °C in an argon atmosphere.

It should be noted that grain boundary embrittlement following high-temperature air exposure or the drastic loss of ductility on air testing at elevated temperatures has also been observed in some superalloys[9,10,11] and pure nickel[12] in the past decade. The intergranular embrittlement of these materials during high-temperature air testing has been attributed to the penetration of oxide down grain boundaries accelerated by applied stress.[13] The intergranular failure after prior air exposure at elevated temperature was shown to be due to oxygen diffusing down grain boundaries and is not connected with any continuous oxide formation.[9] Bricknell and Woodford[12] further proved that the embrittlement on air testing was caused by the embrittled grain boundaries failing in tension, rather than by the stress-accelerated grain boundary oxidation mechanism[14] previously proposed. The mechanisms of intergranular embrittlement for unstressed material following prior air exposure and for air-tested material at intermediate temperatures are thus quite similar.

Since the intergranular brittle failures in superalloys and pure nickel are similar to the "grain boundary pest" problem in intermetallic compounds described above, it would seem reasonable to suggest that the mechanisms of these failures are closely correlated. However, Liu and White[7] have claimed that the mechanism for intergranular embrittlement during high-temperature air testing in boron-doped Ni_3Al is different from that in superalloys. They suggested that oxygen weakens the grain boundary as a result of oxygen adsorption at crack tips rather than by oxygen penetration into the grain boundary through diffusional processes.

In the present study, an ordinarily ductile boron-doped Ni_3Al intermetallic compound was also found to suffer severe intergranular embrittlement after an air exposure at 1200 °C for 100 hours. Using Auger electron spectroscopy (AES), the material failure was analyzed, and a mechanism was proposed to explain this grain boundary embrittlement. The "grain boundary pest" in NiAl described by Seybolt and Westbrook[1] and Turner *et al.*[2]

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and the intergranular embrittlement on stressed Ni_3Al reported by Liu and co-workers[5,6,7] and Taub *et al.*[8] are discussed with reference to this mechanism.

II. EXPERIMENTAL

A boron-doped Ni_3Al intermetallic compound with a nominal composition of Ni-22 at. pct Al-0.6 at. pct Zr-0.2 at. pct B was prepared from Ni and Al of 99.9 pct purity and Ni-B and Ni-Zr master alloys by melting in a vacuum-induction furnace and casting into an iron mold to produce an ingot of 25 × 100 × 80 mm. The ingot was then cut and fabricated into 1-mm-thick sheets by repeated cold rolling (20 to 50 pct) and annealing (1150 °C/2 h and 1000 °C/1 h in air). The impurities included in this alloy were negligible. For example, the total oxygen and carbon contents were less than 60 ppm, the hydrogen content was less than 10 ppm, and the sulfur content was less than 15 ppm by weight. The thermomechanically treated material (TMT) possessed a high ductility of 45 pct elongation according to tensile tests performed at ambient temperature.

Part of the sheet material was polished and further annealed at 1038 °C and 1200 °C in an air furnace for 100 hours. The two sets of air-exposed materials (1038 °C and 1200 °C), as well as the TMT, were cut into small specimens with a diamond saw for Auger analysis. The resultant AES specimens having a size of 3 × 16 × 1 mm were notched at the middle of the two long edges. All of the specimens were ground to 600 grit and cleaned ultrasonically with acetone before Auger analysis. A Physical Electronics Industries Model 600 Scanning Auger System was used to analyze the fracture surfaces. Specimens were fractured *in situ* by impact bending with a hammer at a system pressure of 10^{-9} Pa. In order to promote brittle fracture for some of the more ductile specimens, they were cooled in the AES system with flowing liquid nitrogen before being fractured. A primary electron beam energy of 10 keV was used. The lateral resolution of the AES system was less than 1 μm, and an excellent secondary electron image (SED) could be obtained for the observation of the fracture surfaces. To determine the fracture surface chemistry, the Auger spectra were taken at several points on the surface. Following AES analysis of the as-fractured surfaces, some specimens were sputtered with 3 keV Ar^+ ions using a double-ion gun. The diameter of the ion beam was about 500 μm. The sputtering was performed by scanning of the ion beam over a 3 × 3 mm region. Also, the concentration depth profile beneath the fracture surface was obtained by sputtering in steps followed by Auger analysis. The removal rate of material from the fracture surface by ion bombardment was estimated from the removal rate of atoms from a Ta_2O_5 sputtering standard, which is about 0.5 nm or 2 atom layers per minute for the above operating conditions.

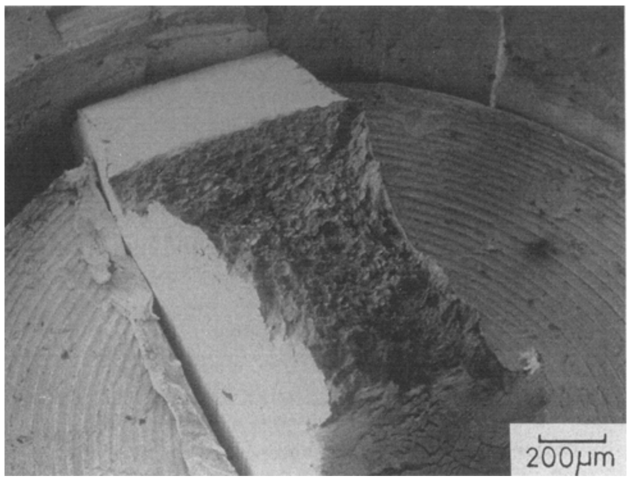
After 1200 °C air exposure, the specimens were also analyzed by energy dispersive X-ray spectroscopy analysis (EDX) with a Be-window in a PHILIPS* 515 scanning electron microscope in order to determine the chemical compositions of the oxide layer and precipitated particles.

*PHILIPS is a trademark of Philips Electronic Instruments Corporation, Mahwah, NJ.

III. RESULTS

A. *TMT Material*

The original TMT specimen shows strong necking behavior after impact bending in the AES unit. Figure 1(a) is a low-magnification SED of the as-fractured AES specimen. The fractography of this material exhibits obvious ductile fracture mode, as shown in Figure 1(b), a high-magnification view of the surface in Figure 1(a). From Figure 1(b), it can be seen that a few portions of the fracture surface exhibit intergranular fracture, with many slip traces existing on the grain boundary facets. The Auger spectrum taken from an intergranular region shows a strong boron peak (Figure 2(a)), while no boron signal was observed from transgranular regions (Figure 2(b)). The absence of embrittling impurities, such



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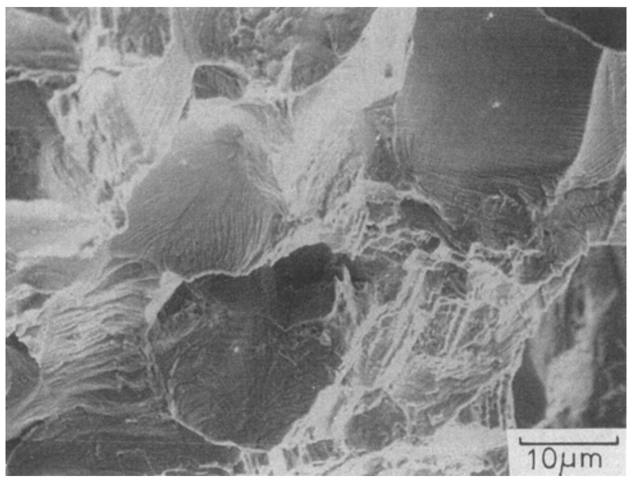


(*b*)

Fig. 1—(*a*) Thermo mechanically treated $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy without long-term air exposure. (*b*) Higher magnification view of (a). (SED of the fractured AES specimen.)

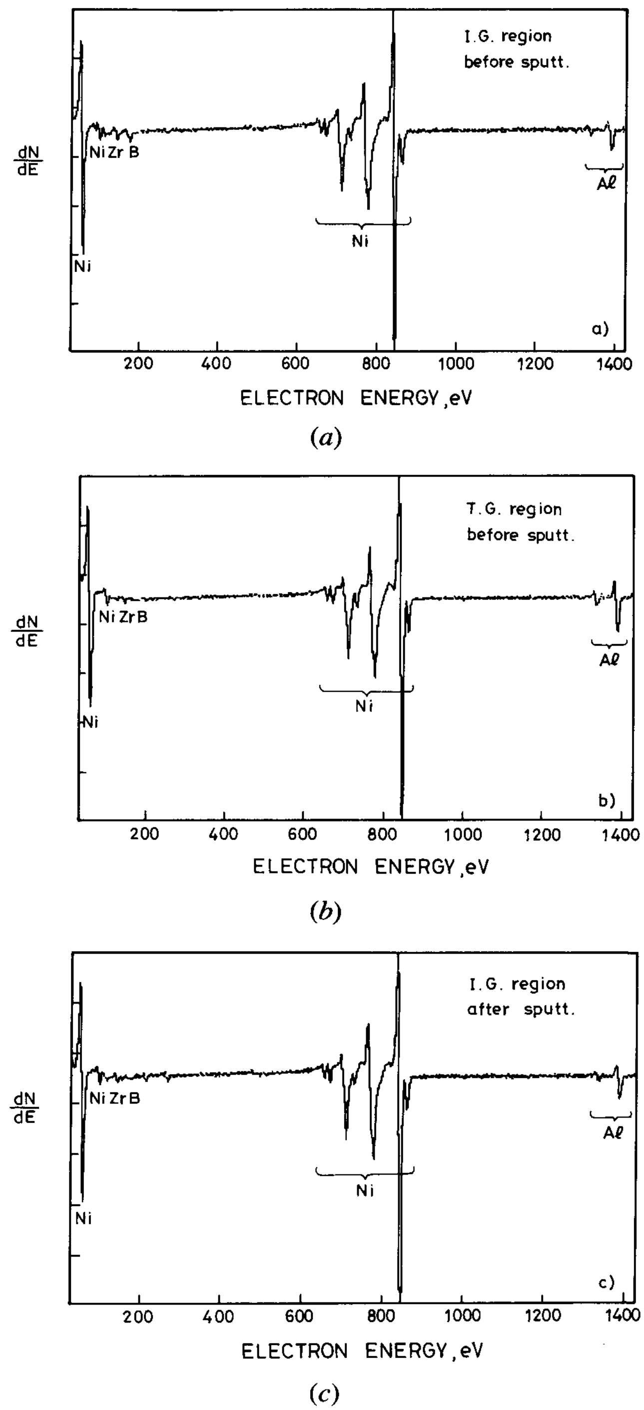


Fig. 2—Auger spectrum taken from (*a*) an intergranular region, (*b*) a transgranular region, and (*c*) an ion-sputtered intergranular region of the fracture surface in Fig. 1.

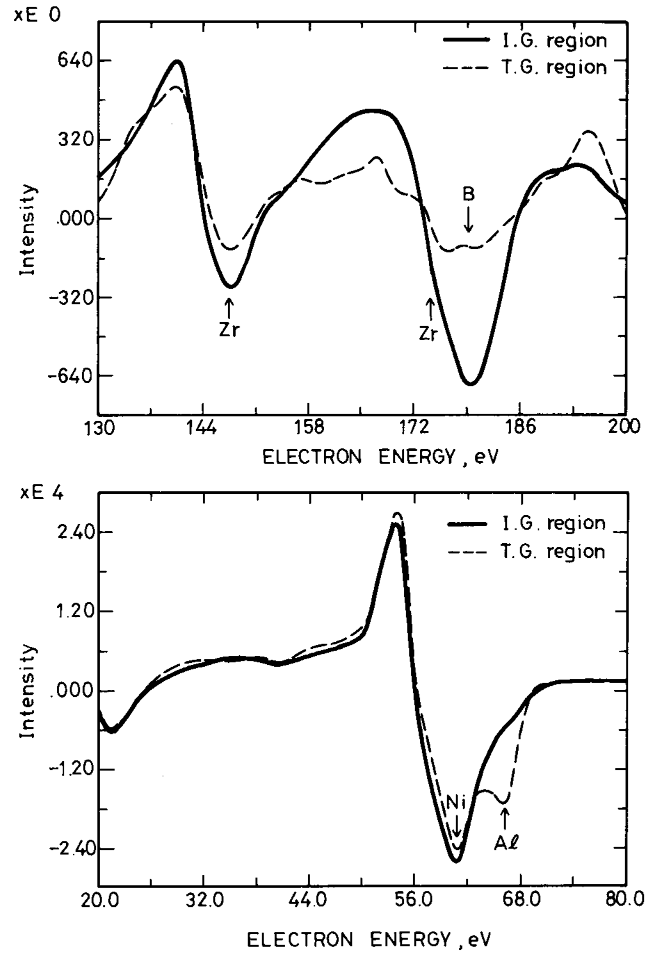


Fig. 3—Auger multiplex spectra taken from an intergranular region and a transgranular region of the fracture surface in Fig. 1.

as oxygen, carbon, and sulfur, is also demonstrated by the Auger spectra. The strong enrichment of boron on grain boundaries can be clearly seen from the multiplex spectrum in Figure 3(a). In Figure 3(a), a slight enrichment of zirconium on grain boundaries can also be observed. On the contrary, the multiplex spectrum in Figure 3(b) shows that aluminum is depleted on the grain boundaries in this alloy. The enrichment of boron and zirconium on grain boundaries disappeared after ion sputtering of several monolayers of material from the as-fractured intergranular surfaces (Figure 2(c)). The results indicate that boron and zirconium segregated with equilibrium-type segregation on the grain boundaries of $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy. The equilibrium-type segregation of boron and zirconium elements can be confirmed from an analysis of the concentration depth profiles obtained by ion sputtering. Figure 4 shows the AES concentration profiles for boron, zirconium, aluminum, and nickel with respect to sputtering time and distance from a grain boundary facet. Summarizing the AES results of TMT material, it can be concluded that in this $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy, both boron and zirconium segregated within several monolayers of the grain boundary. Further study has shown that the aluminum depletion is caused by the zirconium segregation rather than by that of boron.[15]

B. *Material Exposed to Air at 1038 °C for 100 Hours*

After air exposure at 1038 °C for 100 hours, the surface of the $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy was covered with a green oxide film. According to the study of Kuenzly and Douglass,[16] the composition of this green oxide film should be NiO. The specimen impact fractured in the AES system also shows a large amount of necking similar to what was observed for the original TMT specimen

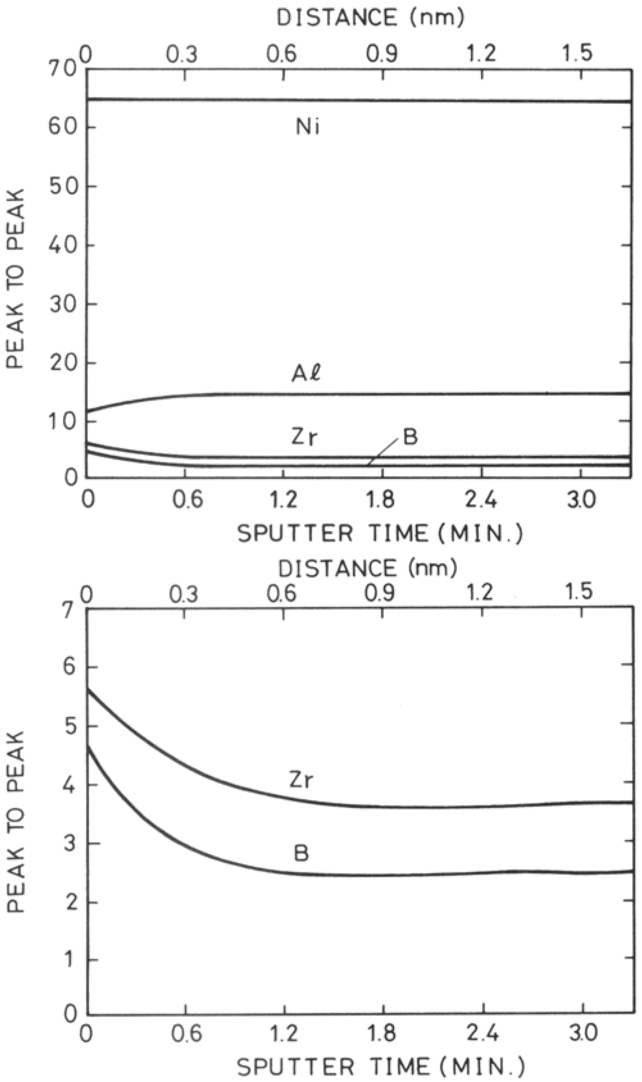


Fig. 4—Concentration profiles of elements with respect to distance from the grain boundary of material not subjected to long-term air exposure.

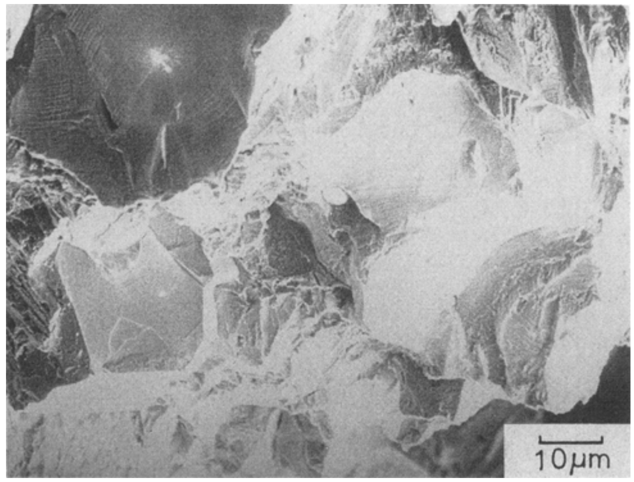


Fig. 5—Fracture surface of $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ after air exposure at 1038 °C for 100 h.

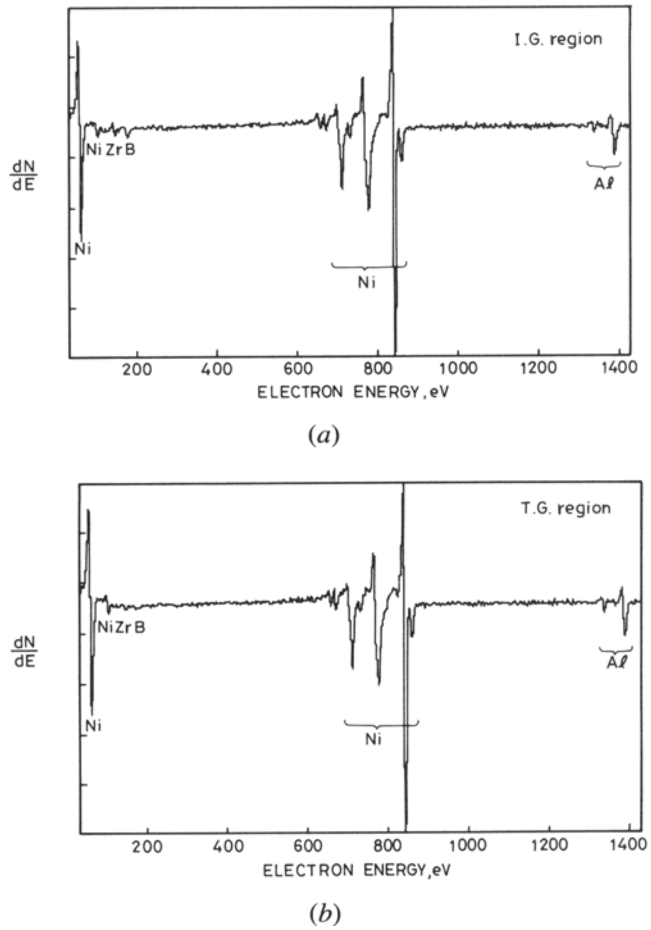
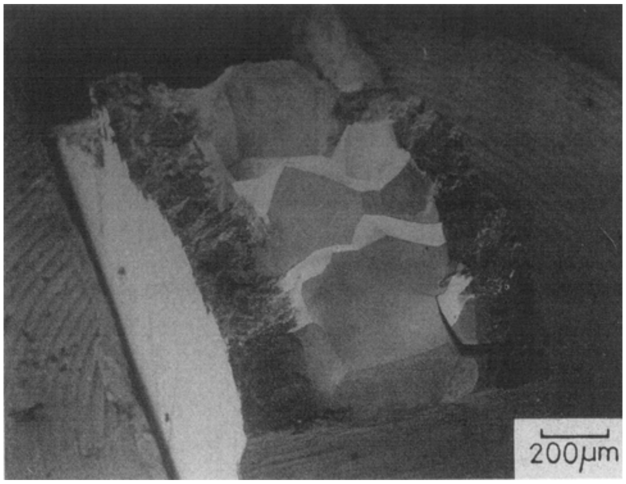


Fig. 6—Auger spectrum taken from (*a*) an intergranular region and (*b*) a transgranular region of the fracture surface in Fig. 5.

(Figure 1(a)). Secondary electron image observation of the fracture surface reveals a ductile fracture mode with few intergranular facets (Figure 5). It can also be seen that many slip traces exist on these intergranular facets. This indicates that even here considerable plastic deformation accompanied the intergranular fracture. By comparing the Auger spectra taken from an intergranular region (Figure 6(a)) with that taken from a transgranular region (Figure 6(b)), the segregation of boron on grain boundary can also be demonstrated. It should be especially noted that even after long-term air exposure at 1038 °C for 100 hours, no oxygen, carbon, or sulfur can be found in the Auger spectra. This evidence may be associated with the unchanged, excellent ductility of this material.

C. *Material Exposed to Air at 1200 °C for 100 Hours*

After air exposure at 1200 °C for 100 hours, the oxide film of this material became black. The outward appearance of the fractured AES specimen was also quite different from that observed on the original TMT and 1038 °C air-exposed materials. No necking is observed on the fractured AES specimen (Figure 7(a)). Both sides of the specimen in Figure 7(a) were converted to a thick layer of oxidation product. The remaining material in the



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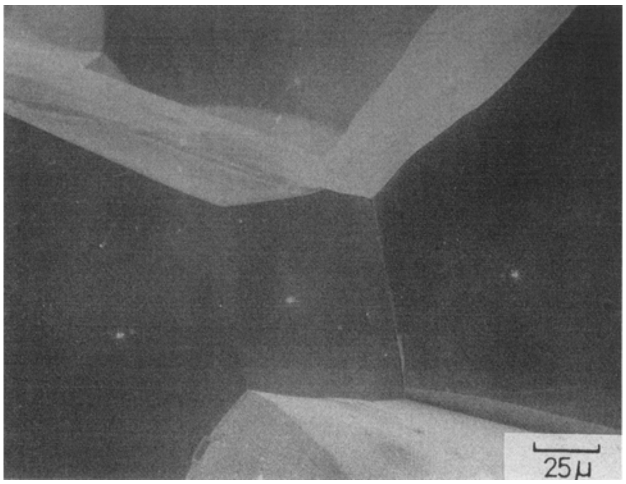


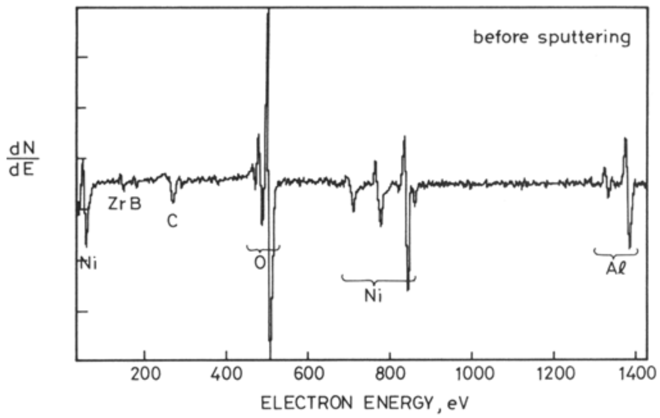
(*b*)

Fig. 7—(*a*) A $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy after air exposure at 1200 °C for 100 h. (*b*) Higher magnification view of (a). (SED of the fractured AES specimen.)

interior of the AES specimen shows completely intergranular fracture. In fact, the ductility of the material in this case has almost totally vanished. On the intergranular facets, many oxide particles can be found. And the slip traces on the fracture surfaces of the TMT material and that treated at 1038 °C are absent (Figure 7(b)). Further metallographic analysis showed that there also exist many oxide particles in grain interiors. The occurrence of oxide particles in grain interiors has also been reported by Turner *et al.*[2] in their work on grain boundary pest in NiAl alloys.

An interesting phenomenon which should also be noted is the enormous grains observed in the 1200 °C air-exposed specimen. By comparing the grain sizes before and after the 1200 °C air exposure, it can be shown that grains grow dramatically from 20 to 250 μm in diameter. This somewhat unexpected abnormal grain growth during air exposure at 1200 °C was also reported by Bricknell and Woodford in an iron-base superalloy.[12] Still it is quite surprising that the ratio of grain growth in the present study is very consistent with that which was observed by Bricknell and Woodford, if we consider that the grain boundaries in our thinner AES specimens, as in Figure 7(a), were attacked by oxygen from both sides. In the work of Bricknell and Woodford, abnormal grain growth was observed on the external region of their thicker specimens.[12] For the grain boundaries under investigation in their case, oxygen was transported from only one side of the specimens.

The most significant result linked to the severe intergranular embrittlement can be deduced from the Auger spectra shown in Figure 8. Figure 8(a) shows that a very large amount of oxygen is located on the as-fractured surface, and carbon contamination on grain boundaries can also be seen in the Auger spectrum. For a few grain boundary facets, the zirconium peaks at 147 eV have been observed to be somewhat larger, which could have resulted from the attraction of the zirconium from the grain interiors to the grain boundaries by the oxygen segregated on the grain boundaries. In addition to the appearance of these impurity peaks, the aluminum peak at 1396 eV has shifted slightly. This indicated that the aluminum was transformed into aluminum oxide. It should also be noted that the boron peak still exists in spite of the enormous enrichment in oxygen. Figure 8(b) shows the Auger spectrum after ion sputtering for 3 minutes. Analysis of the spectrum indicates that the enrichment of the contamination elements, oxygen and carbon, disappears totally after the removal of several monolayers



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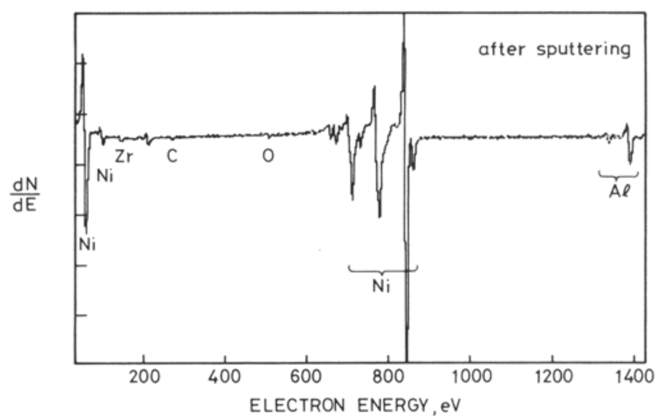

(*b*)

Fig. 8—Auger spectrum taken from (*a*) the as-fractured surface and (*b*) the ion-sputtered surface of the fracture surface in Fig. 7.

of material from the intergranular facets, and the spectrum returns to that obtained for the TMT specimen. Also, the shifted aluminum peak returns to its original positions. The Auger depth profile in Figure 9 shows that the concentrations of oxygen and carbon decrease rapidly within a distance of about 10 Å. This proves that these contamination elements segregated on the grain boundaries in an equilibrium fashion. In Figure 9, it can also be seen that the aluminum concentration remains constant with respect to distance from the grain boundary, while nickel is strongly depleted on it.

A further comparison of the surface concentration of oxygen for the internal intergranular fracture region and the external oxidation layer region (Figure 10(a)) was also performed to evaluate the distribution of oxygen in the latter material. The Auger spectra in Figures 10(b) and (c) indicate that the surface oxygen content in the external oxide layer is evidently lower than that on the internal intergranular fracture surface. However, the EDX analysis, which gives the concentration of elements within a depth of about 10^4 Å, shows an opposite result. Comparing the oxygen peaks in the two EDX diagrams, which

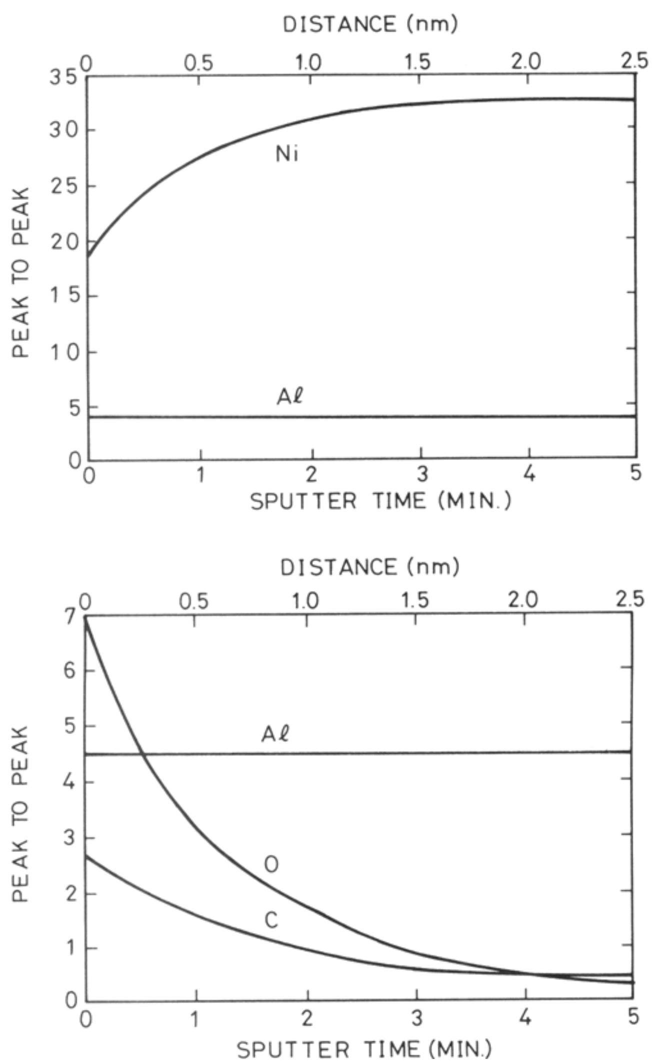

Fig. 9—Concentration profiles of elements with respect to distance from the grain boundary shown in Fig. 7.

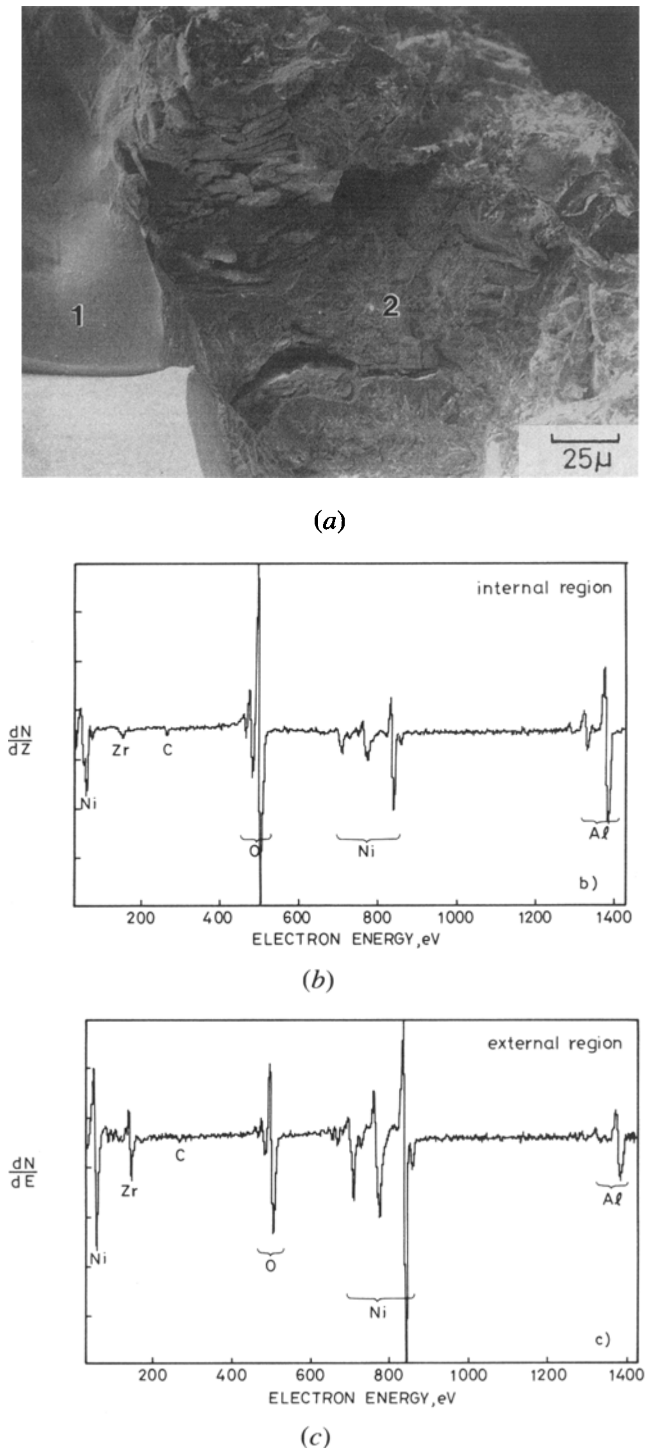

Fig. 10—(*a*) Morphology of fracture surface in the internal intergranular region (1) and external oxide layer region (2), (*b*) Auger spectrum taken from the internal intergranular region, and (*c*) Auger spectrum taken from the oxide layer region.

were taken from the internal intergranular and external oxidized regions, respectively, shows that the external oxide layer possesses a higher level of oxygen (Figure 11(a)) than the internal intergranular region (Figure 11(b)). Summarizing the above AES and EDX results, it can be said that the oxygen content on the

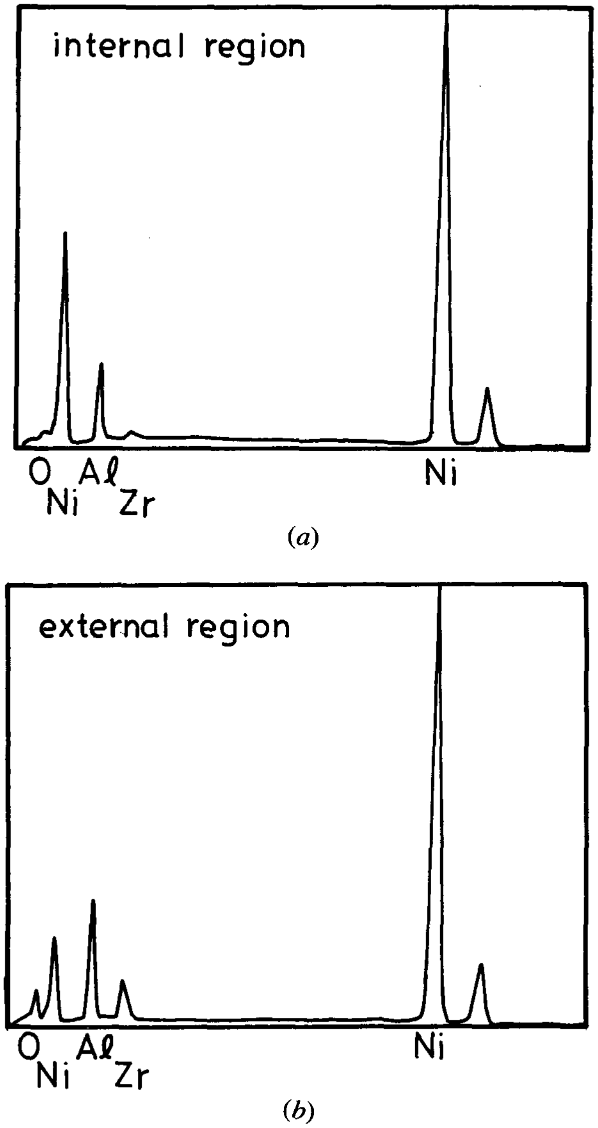

Fig. 11—EDX analyses of (*a*) the internal intergranular region and (*b*) the external oxide layer region of Fig. 10(a).

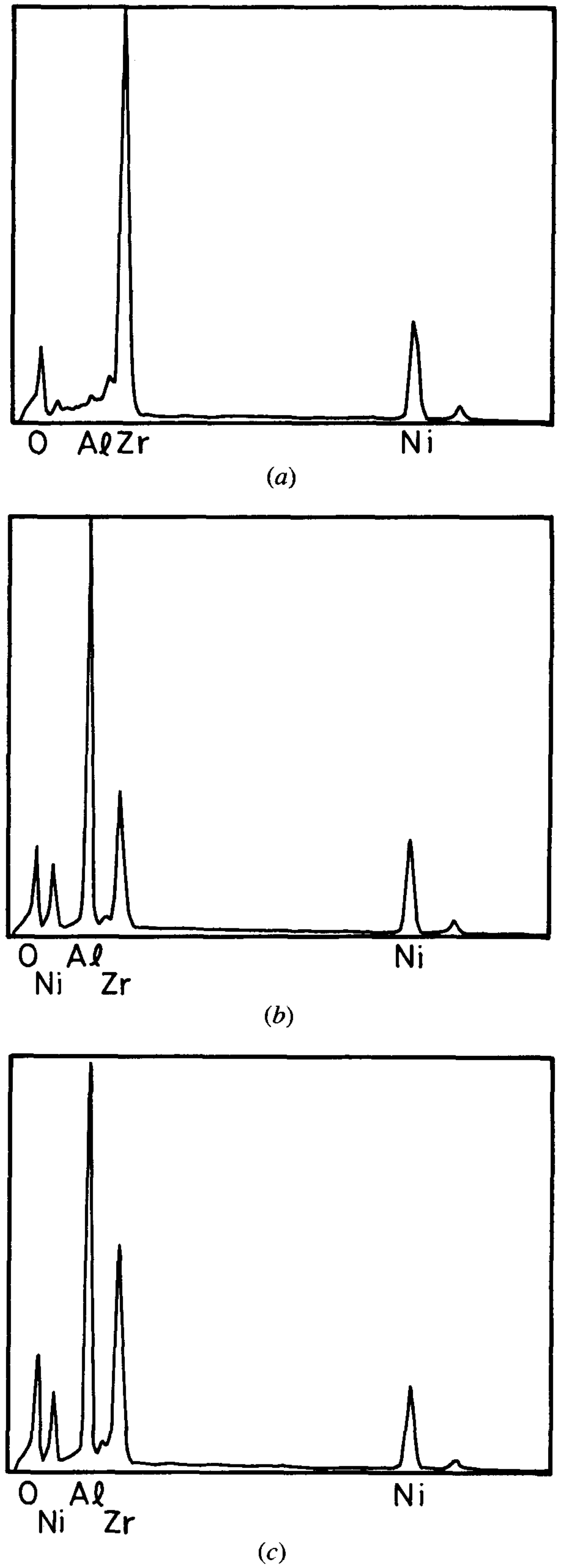

Fig. 12—(*a*) through (*c*) EDX analyses of oxide particles on grain boundaries showing the compositions characteristic of ZrO, Al_2O_3, and $ZrAl_2O_4$, respectively.

grain boundary is far in excess of that in the oxide film on the surface of this material. And the oxygen concentration in the internal grain is lower than that in the oxide film.

The oxide particles as analyzed by EDX show various compositions. A comparison of the EDX diagrams from the particles with that taken from the grain boundary itself (Figure 11(b)) suggests that the oxide particles are ZrO, Al_2O_3, and $ZrAl_2O_4$ (Figures 12(a) through (c), respectively). No NiO particles could be found on grain boundaries. However, EDX analyses of the particles contained in the external oxide layer indicate the presence of NiO in addition to the above three compounds.

IV. DISCUSSION

By trace alloying of the base $Ni_{77.4}Al_{22}Zr_{0.6}$ intermetallic compound with 0.2 at. pct B to form the intermetallic compound $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$, its room-temperature ductility was drastically improved. The beneficial effect is attributed to the equilibrium segregation of boron on grain boundaries, as shown by AES analyses. However, after air exposure at 1200 °C for 100 hours, the material suffered severe intergranular embrittlement. This type of material damage has been dubbed "grain boundary pest," and its mechanism was discussed extensively during the 1960s. In the present study, AES analyses showed enormous oxygen segregation on grain boundaries of the alloy exposed to air at 1200 °C, and many particles of ZrO, Al_2O_3, and $ZrAl_2O_4$ were found on fracture surfaces. The oxygen segregation to the grain boundary is consistent with the embrittlement theory suggested by Seybolt and Westbrook.[1] On the other hand, the existence of precipitated particles on grain boundaries is consistent with the observations of Turner *et al.*[2] Turner *et al.* also reported that the "grain boundary pest" problem in NiAl occurred only at about 1400 °C. In the present study, the $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ allow showed no evidence of intergranular embrittlement after air exposure at 1038 °C for 100 hours. The Auger spectrum of the fracture surface of the alloy exposed to air at 1038 °C remained the same as that for the TMT material without air exposure. The present results imply that grain boundary pest in Ni_3Al also occurs only above a certain elevated temperature of approximately 1200 °C.

By linking the observations of oxygen segregation, oxide particles, and temperature to the observed severe grain boundary failure, a modified mechanism explaining the "grain boundary pest" problem of Ni_3Al-type intermetallic compounds is proposed. According to this mechanism, the catastrophic embrittlement is caused by the disruption of the protective oxide film on the alloy surface and oxygen penetration into and segregation on its grain boundaries. At lower exposed temperatures, an adherent and compact oxide film forms on the surface of nickel aluminide-type intermetallic compounds. This oxide film protects the alloy against penetration of oxygen into the material, and intergranular embrittlement does not occur. Above a certain elevated temperature, the oxide film is disrupted, and this allows the oxygen from the air atmosphere to diffuse into and along the grain boundaries and to segregate on them in a short period of time. For alloy specimens tensile tested in air, the disruption of the protective oxide film may be aided by the applied stress, and the temperature to allow the penetration of oxygen into grain boundaries and cause intergranular embrittlement is thus lower. In this manner, the intergranular embrittlement observed by Liu and White[7] and Taub *et al.*[8] can be explained. The trace amounts of residual oxygen contained in the 7.5×10^{-6} torr vacuum used by Liu and White[7] and in the argon atmosphere used by Taub *et al.*[8] are thus thought to be responsible for the grain boundary damage in the alloys used in these studies. The higher oxygen content in the argon atmosphere is believed to have resulted in a lower temperature for occurrence of the intergranular embrittlement. In the work of Liu and White,[7] they also reported that the gage section of their specimens after air testing at 600 °C remained ductile at room temperature. Because the gage section of the specimens during the 600 °C air testing was subjected to negligible tensile stresses, the prerequisite temperature for the occurrence of grain boundary embrittlement was approximately 1200 °C according to the present mechanism. The survival of this gage section at 600 °C in the work of Liu and White[7] is thus conceivable. In a recent work, referencing to an observation of Choudhury *et al.*[17] on the effect of temperature on boron segregation, Takeyama and Liu[18] suggested that the high-temperature embrittlement of boron-doped Ni_3Al is, at least partially, attributed to the thermal variation of boron segregation on grain boundaries. This may, to some degree, be responsible for the intergranular failure of boron-doped Ni_3Al during tensile testing in air at intermediate temperatures. However, for unstressed boron-doped Ni_3Al like that of the present study, the grain boundary embrittlement after prior air exposure at elevated temperature cannot be linked to the variation of boron segregation, because the specimens were fractured at ambient temperature. In fact, the present Auger analyses (Figure 8(b)) have indicated that the boron segregation did not change in spite of the relatively high oxygen content on the grain boundary. Also, the boron variation cannot explain the greatly different fracture behavior between the specimens exposed to air at 1038 °C and 1200 °C.

The anomalous grain growth observed in the $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy after air exposure at 1200 °C is consistent with the observations reported by Bricknell and Woodford[12] in an iron-base superalloy. They found that the grain size of an IN903A* superalloy increased from 50 to 300 μm after air exposure at 1000 °C for 100 hours. In the present $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ intermetallic compound, the grain size increased from about 20 to 45 μm after exposure to air for 100 hours at 1038 °C, while it increased to 250 μm after 100 hours at 1200 °C. This implies that the anomalous grain growth in the present study is correlated to the effect of oxygen on the grain boundaries.

Bricknell and Woodford proposed that oxygen reacted with the carbon contained in their iron-base superalloy to form carbon dioxide and monoxide and that this resulted in the release of the original solute pinning effect of the carbon on the grain boundaries. Since no carbon is contained in the present $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy, the above explanation cannot, however, be adapted to the present study.

In order to explain the anomalous grain growth, another unpinning effect on the grain boundary migration in the present alloy is considered. It has been known that a Ni_3Al-base alloy with Ni content higher than 76.5 at. pct is usually possessed a Ni-riched second phase on the grain boundaries. In the present study, 77.4 at. pct Ni is contained in the Ni_3Al-base alloy. The presence of grain boundary second phase in the alloy acts as a pinning source on its grain boundary migration during the grain growth. The segregation of oxygen on grain boundaries after long-term air exposure at 1200 °C results in a drastic decrease in nickel contents at grain boundaries, which has been shown in Figure 9. The depletion of nickel causes the dissolution of Ni-riched second phase at grain boundaries, which leads to the reducing

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or even disappearance of the pinning effect on grain boundary migration. The grains can thus grow in an abnormal manner.

V. CONCLUSIONS

$Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ intermetallic compound exhibited excellent ductile fracture behavior due to equilibrium boron segregation on grain boundaries. After 1038 °C of air exposure for 100 hours, the material remained ductile. The Auger spectrum from the fracture surface showed no difference from that of the original untreated material. However, the material suffered severe intergranular embrittlement after 100 hours of air exposure at 1200 °C. Many oxide particles were found on the grain boundaries of this material. Auger electron spectroscopy analyses showed a strong oxygen segregation on grain boundaries, while boron segregation was not changed. The oxygen segregation was also limited within the depth of several monolayers from the grain boundary. To explain this intergranular embrittlement, a modified mechanism was proposed according to which the protective oxide film is disrupted above a certain elevated temperature approximately 1200 °C. This allows the oxygen in the atmosphere to diffuse into and along the grain boundaries and to segregate on them. The oxygen segregated at grain boundary can also oxidize Al and Zr to form various oxide particles such as ZrO, Al_2O_3, and $ZrAl_2O_4$. Their existence has been confirmed by EDX. The present mechanism can be adapted to explain the dramatic intergranular fracture observed during tensile testing in air at elevated temperatures. Finally, an anomalous grain growth was found to accompany the "grain boundary pest." The grain size increased from about 20 to 250 μm after being exposed to air at 1200 °C for 100 hours, in comparison with the normal grain growth from 20 to 45 μm observed during the 100-hour air exposure at 1038 °C. The anomalous grain growth is consistent with the observations of Bricknell and Woodford[12] of a similar grain growth in an iron-base superalloy. The solute unpinning effect of the carbon on the grain boundaries by reacting the penetrated oxygen has been proposed by Bricknell and Woodford to explain this phenomenon. In this work, it is suggested that the strong depletion of nickel on grain boundaries, due to the segregation of oxygen, results in the dissolution of Ni-riched second phase presented on the grain boundaries of this $Ni_{77.4}Al_{22}Zr_{0.6}B_{0.2}$ alloy. The original pinning effect of the second phase on the grain boundary migration is thus removed, which leads to the anomalous grain growth in this alloy after long-term air exposure at 1200 °C.

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