Investigation of the heat-affected zone fracture in Ni_3Al welds

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Ternary $Ni_{77}Al_{23-y}X_y$ (X = Zr or Hf, $y = 0.5$ or 1) + 500 ppm boron compounds with various grain sizes were welded by a CO_2 laser. Fractographic examinations of the heat-affected zone (HAZ) in the welds with or without postweld heat treatment (PWHT) were performed on the impact-fractured specimens. In laser welds, the fracture appearance of the HAZ was mixed transgranular/intergranular modes for fine-grained alloys and intergranular mode for coarse-grained materials. However, an entirely transgranular mode was observed in the base metal regardless of the grain size of the compounds. Boron desegregation at high temperatures during the thermal cycle of welding could be used to explain the fractographic change from originally ductile mode into less-ductile or even brittle fracture in the HAZ. Short-term PWHTs along with slow cooling provided sufficient time for boron segregation back to the grain boundary, resulting in a completely transgranular fracture mode in the fine-grained HAZ. Nevertheless, such a phenomenon was not observed in the HAZ of coarse-grained welds. Cracks in the HAZ of coarse-grained welds after long-term PWHT, if not so severe, could be healed by a sintering process.

I. INTRODUCTION

Investigations of Ni_3Al intermetallic compounds as high temperature structural materials have been of great interest in recent years. Ni_3Al sustains long-range order structure up to its melting temperature, exhibits unusual temperature dependence on yield strength,[1,2] and excellent oxidation resistance.[3] Previous studies have indicated that the inherent grain boundary brittleness of Ni_3Al impedes the applicability of Ni_3Al in polycrystalline form,[4] whereas the addition of boron in trace amounts significantly improves the ductility of this compound.[4,5]

For structural applications, weldability is an important consideration in the developing process of a material. Recent works indicated that HAZ cracking is the main problem associated with the welding of Ni_3Al.[6–10] The reduction of boron addition (optimum amount 200 ppm) in Ni_3Al and the decrease of welding speed have been reported to effectively overcome this problem in electron beam welds.[6] The addition of Hf to a Ni_3Al + 200 ppm boron alloy has been found to improve resistance to HAZ cracking.[8,9] Our previous study also indicated that grain refinement is beneficial to the weldability of Ni_3Al with various boron contents.[10] To maintain good weldability, the control of grain size below a certain critical value is very important. Although the emphasis in these studies has been on the elimination of the HAZ cracking, the main cause of HAZ cracks is still not clear.

Choudhury *et al.*[11] reported that slow cooling promotes grain boundary segregation of boron in Ni_3Al, while rapid quenching retains the low boron level at high temperatures. It was also found that the effect of boron on the improvement of Ni_3Al ductility disappears above a critical temperature range.[12] It is implied that grain boundary weakness resulting from boron desegregation at elevated temperatures might occur during the welding process of Ni_3Al alloys. Therefore, the aim of this study was to investigate the characteristics of HAZ fracture in the compounds and the behavior of boron in the welding process. A series of fractographic observations and compositional analyses were performed on Ni_3Al laser welds with various ternary additions and grain sizes. In addition, postweld heat treatments (PWHTs) were also conducted on some specimens to correlate the metallurgical response in the HAZ of Ni_3Al welds.

II. MATERIALS AND EXPERIMENTAL PROCEDURE

Ternary compounds with the chemical composition (at. %) $Ni_{77}Al_{23-y}X_y$ (X = Zr or Hf, $y = 0.5$ or 1) + 500 ppm boron were used in this study. The identification number, chemical composition, and grain size of the specimens are listed in Table I. Commercially pure Ni and Al (both with 3 N purity) together with high purity Ni–Zr (1 : 1), Ni–Hf (1 : 1), and Ni–B (4 : 1) master alloys were vacuum-induction melted to obtain ingots. Ingots were sliced and then subjected to thermomechanical treatment (TMT) by repeated cold rolling, with intermediate annealing at 1000 °C or 1050 °C, into strips of 1.2 mm. The amount of deformation and the final annealing temperature/time were adjusted to

TABLE I. Identification number, chemical composition, and grain size of the specimens used in this study.

Specimen I.D.	Ni (at. %)	Al (at. %)	Zr (at. %)	Hf (at. %)	B (wt. ppm)	Grain size (μm)
B3-8						8
B3-16	77	22	1	...	500	16
B3-26						26
B5-100	77	22.5	0.5	...	500	100
C1-8						8
C1-16	77	22	...	1	500	16

generate various grain sizes. All specimens had a homogeneous microstructure of gamma prime phase ($L1_2$ ordered structure) with equiaxed grains after the TMT process. The grain sizes were determined by using the linear intercept method.

For the laser welding experiments, a commercially available CO_2 laser (Spectra-Physics 825) was employed to perform high speed autogenous welding. Bead-on-plate welds were made with a fixed welding speed of 203 cm/min, and the laser power (~2000 W) was adjusted to achieve full penetration welds. PWHTs were performed at 1000 °C for various holding times of up to 75 h and followed by furnace cooling (F.C.). To prevent severe oxidation, the welds for long-term PWHTs were encapsulated into a quartz tube with high purity argon, whereas the specimens for short-term PWHT were exposed directly in an air furnace. All welds with or without PWHT were sectioned for metallographic examinations. Fractographic studies of the HAZ were made by impact fracture of the U-notched specimens in a direction perpendicular to the welding direction and examined with a scanning electron microscope (SEM).

III. RESULTS

Figure 1 is an optical micrograph of a B3-8 weld. The base metal of the compound, at the right half of the figure, revealed fine equiaxed grains after TMT, while the fusion zone at the left showed a columnar structure. It is important to note that neither grain growth nor intergranular cracking was found in the HAZ of the weld. Figures 2(a)–2(c) are SEM fractographs of the impact-fractured B3-8 weld showing the fusion zone, the base metal, and the HAZ, respectively. Figure 2(a) shows a typical ductile fracture with dendritic solidification feature of the fusion zone in Ni_3Al welds. The region of the heat-unaffected base metal [Fig. 2(b)] exhibited an entirely transgranular fracture mode. Such a ductile feature can be attributed to the beneficial effect of boron segregation to the grain boundaries.[4,5] The HAZ fractograph [Fig. 2(c)] displayed intergranular/transgranular fracture modes, in which slip traces were observed on some intergranular surfaces. Fractographic examinations of the B3-16, C1-8, and C1-16 welds were similar to those of the B3-8 weld. In addition, metallographic observations of these fine-grained welds did not reveal any intergranular cracking in the HAZ.

For coarse-grained materials, the metallographic and fractographic characteristics in the HAZ are different from those of the fine-grained welds. Figure 3 is an optical micrograph of the B3-26 weld showing intergranular cracks in the HAZ. Figure 4 is the fracture surface of a coarse-grained weld after impact loading, in which the change of fracture mode in various regions is obvious. The fracture appearance in the HAZ showed a completely intergranular fracture mode, which is different from the mixed intergranular/transgranular modes of fine-grained welds. Figure 4 also reveals the presence of liquid films on the intergranular surfaces in the HAZ, which was not observed in the fine-grained welds. The liquated region was located mainly in the vicinity of the fusion boundary and extended approximately 4–5 grain diameters into the HAZ. The amount of liquid films in the HAZ decreased as the distance from the fusion boundary increased. Figure 5 displays liquid films on the intergranular surface with droplet-like appearance. Energy dispersive x-ray (EDX) analyses (Fig. 6) of the droplet-like phases coincided, in general, with those

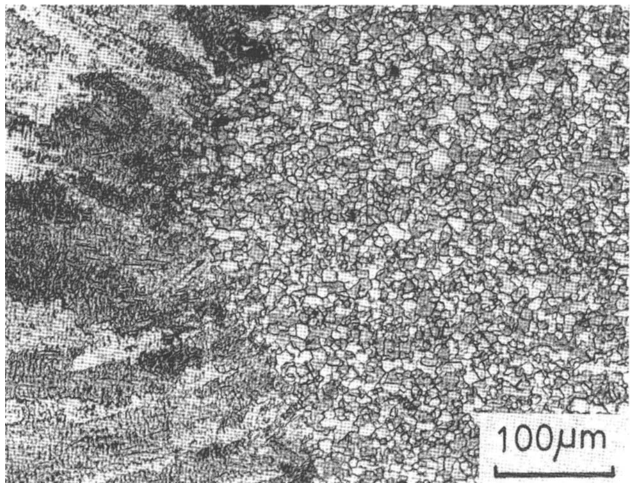

FIG. 1. Optical metallograph of the B3-8 weld showing no intergranular cracks in the HAZ.

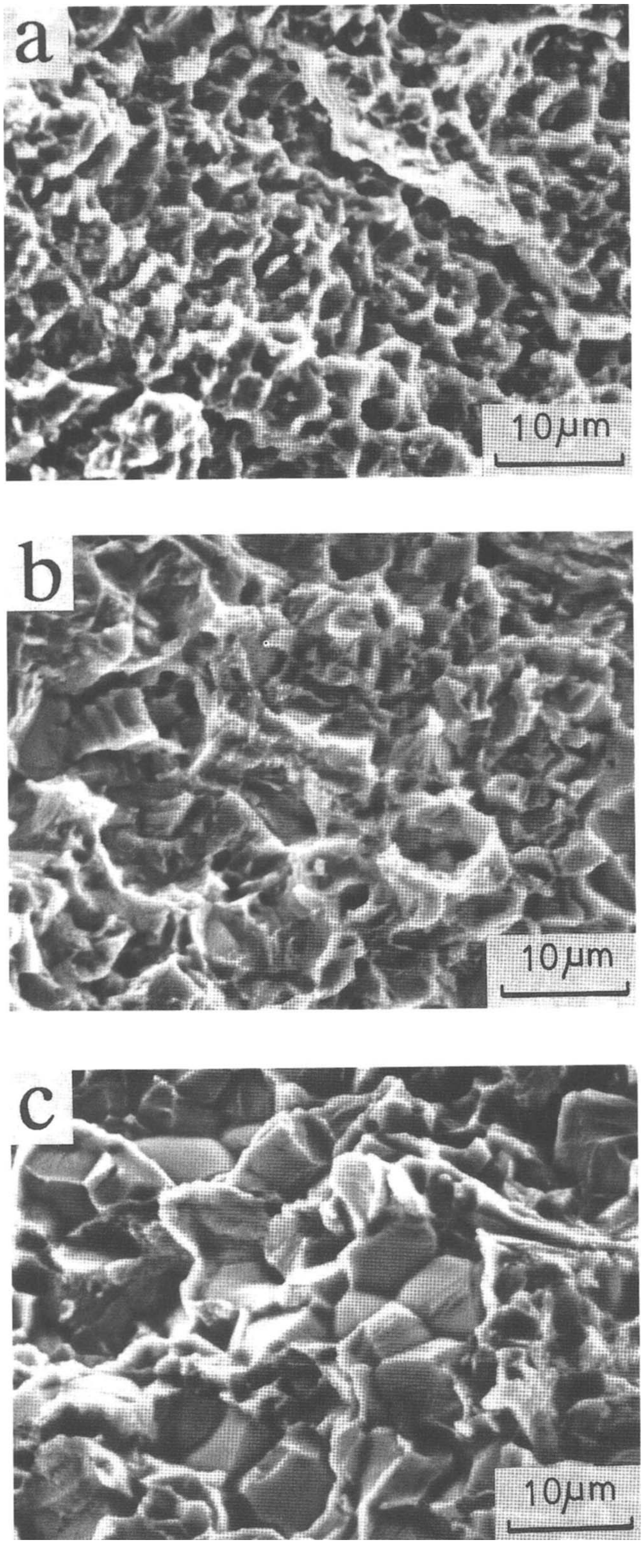

FIG. 2. SEM fractographs showing (a) the fusion zone, (b) the heat-unaffected base metal, and (c) the HAZ of the B3-8 weld.

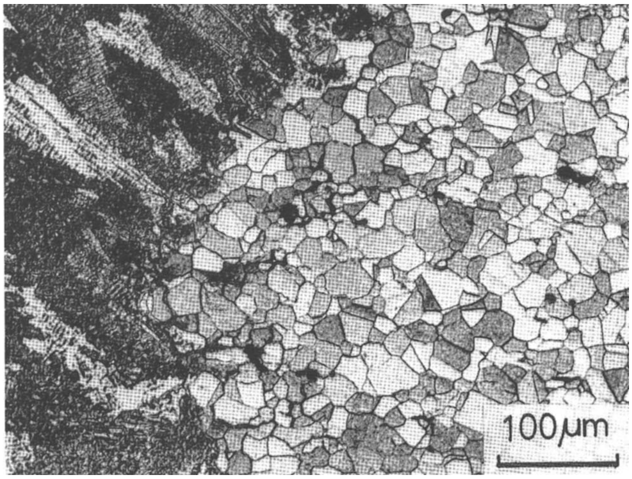

FIG. 3. Optical micrograph of the B3-26 weld showing the presence of the HAZ cracks.

of the gamma prime and gamma (Ni solid solution, disordered fcc structure) phases. Furthermore, a few feathery-like products (indicated by arrows) were also observed, and they were enriched in Zr and impoverished in Al relative to the smooth intergranular surfaces, as shown in Fig. 7. According to the Ni–Zr binary phase diagram,[13] the eutectic temperature at which the Ni_5Zr compound formed was relatively low, approximately 1172 °C. It was also found that grain boundary melting, associated with the formation of a low melting Ni_5Zr phase, occurred in the $Ni_{77}Al_{22}Zr_1$ alloy subjected to a 1300 °C/15 min heat treatment.[14] The EDX spectrum shown in Fig. 7(b) was very similar to that of the Ni_5Zr compound. Consequently, the feathery-like products as shown in Fig. 7(a) could be such a compound. Figure 8(a) shows the fracture surface of the B5-100 weld, in which the molten metal was spread along intergranular paths in proximity to the fusion boundary [Fig. 8(b)]. Chemical analyses of liquid films in the HAZ of B5-100 weld (less Zr content) were similar to those of the B3-26 weld, except less Ni–Zr compound was observed.

When the PWHT was performed on fine-grained welds (B3-8, B3-16, C1-8, and C1-16), the HAZ fracture showed a significant change in appearance as compared to the as-welded conditions. Figures 9(a) and 9(b) illustrate the fracture mode in the HAZ of the C1-16 weld before and after PWHT, respectively. The HAZ fracture was mainly transgranular for the weld after PWHT of 1000 °C/15 min followed by furnace cooling [Fig. 9(b)]. Mixed fracture modes were observed in the HAZ of the welds in the as-welded condition [Fig. 9(a)]. When the holding time of PWHT was prolonged to 30 min, there were no further changes in HAZ fractography of the welds. Metallographic observations revealed that short-term PWHT caused only slight grain growth in the base metal and the HAZ, but did not cause any significant change in the structure of the fusion

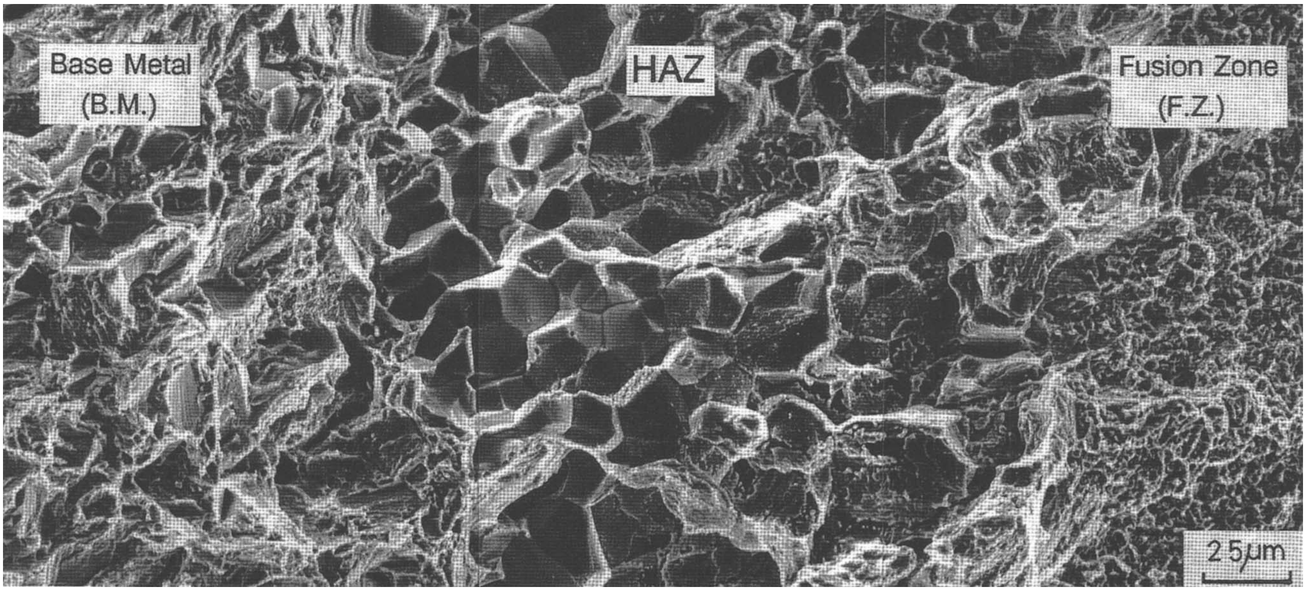

FIG. 4. Impact-fractured surface of the B3-26 weld showing the fracture modes in various regions. Note the presence of liquid films in the HAZ adjacent to the fusion boundary.

zone. For the B3-26 weld that exhibited HAZ intergranular cracks, PWHT at 1000 °C/15 min did not alter the fracture appearance of the HAZ. The metallographic and fractographic characteristics of the weld changed noticeably when the holding time of the PWHT was increased. Figure 10 is an optical micrograph of the B3-26 weld after PWHT of 1000 °C/75 h. Clearly, long-term heat treatment caused significant grain growth in the weld. Furthermore, the fusion zone that originally exhibited columnar-grained structure was changed to a single-phase (gamma prime) structure of equiaxed grains, similar to the structure in the base metal and the HAZ. Accordingly, the same fracture (transgranular) mode was observed for all regions in the weld after long-term PWHT. It is important to note that intergranular cracks in the HAZ were also healed after prolonged PWHT. Crack healing and fracture mode change after PWHT of 1200 °C/5 h were also found in the B3-26 weld.

IV. DISCUSSION

In the present study, laser welded $Ni_{77}Al_{22}X_1$ (X = Zr or Hf) compounds having fine grain sizes showed no significant difference between the HAZ and the base metal in the metallograph. However, the fracture modes of the welds in the as-welded condition

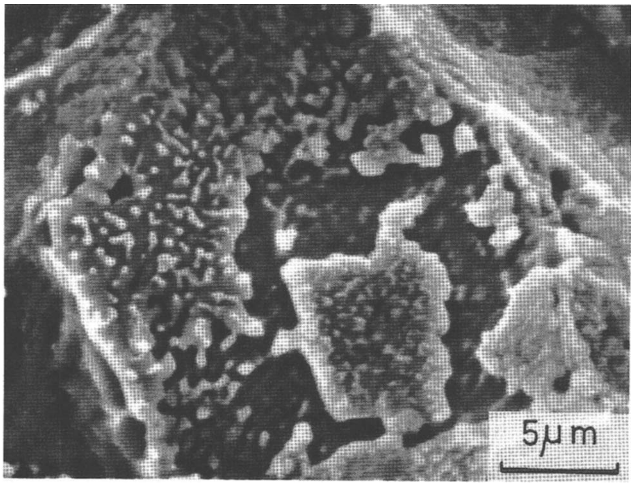

FIG. 5. An enlarged view of the liquated HAZ showing liquid films on the intergranular surface.

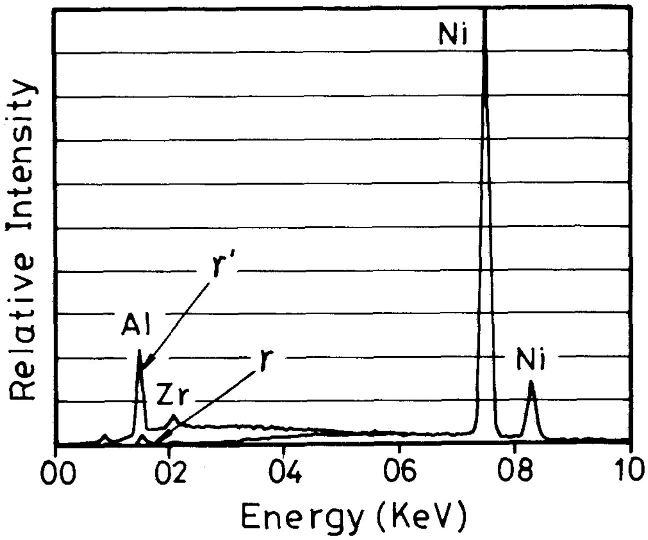

FIG. 6. EDX spectra of the liquated region as shown in Fig. 5.

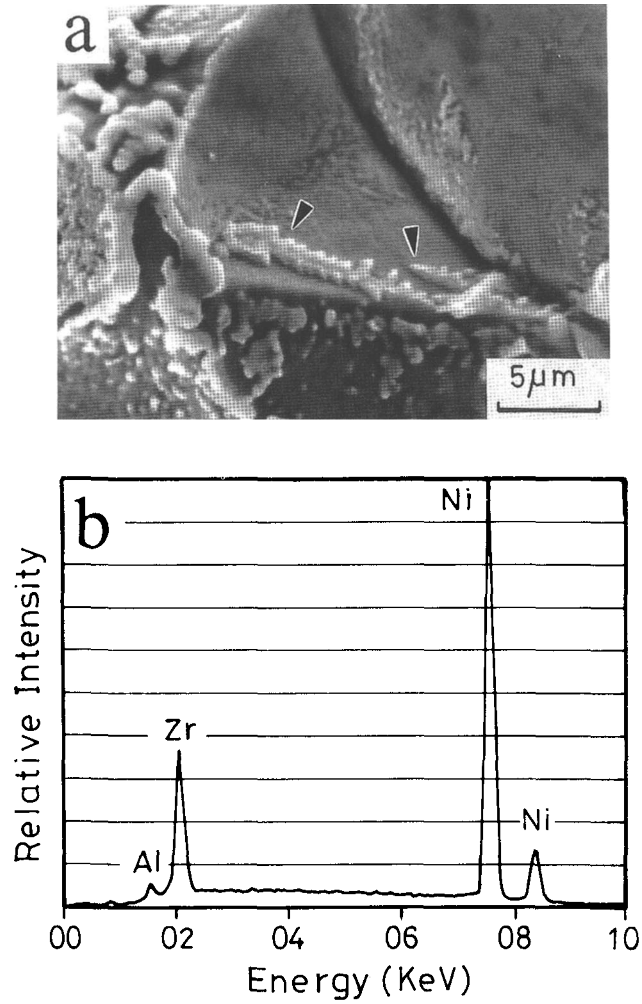

FIG. 7. (a) SEM fractograph and (b) corresponding EDX spectrum for the feathery-like products (indicated by arrows) in the liquated region.

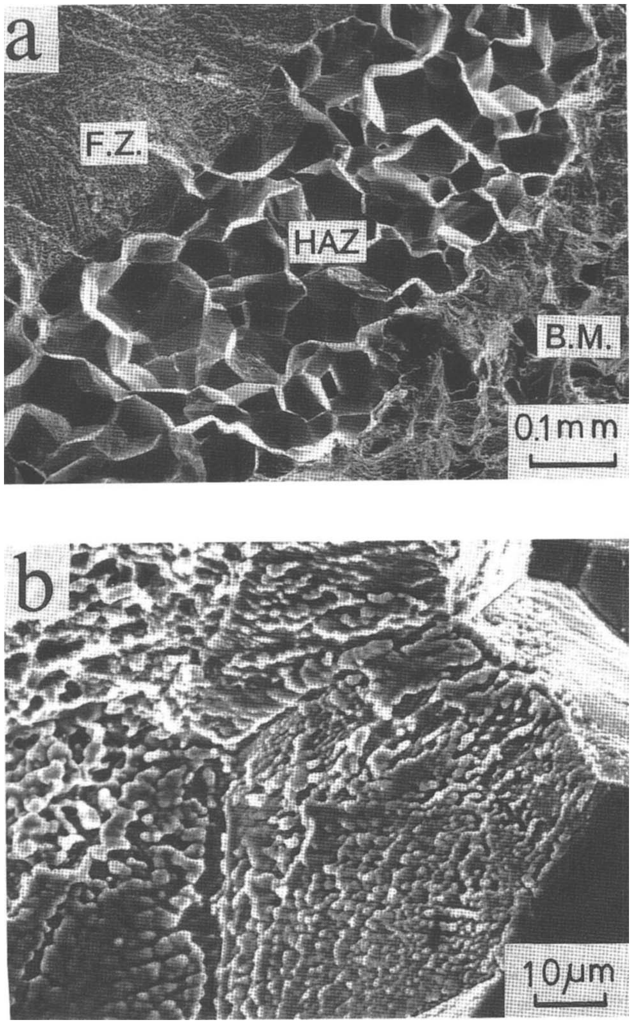

FIG. 8. SEM fractographs of the B5-100 weld showing (a) the impact-fractured surface and (b) an enlarged view of the HAZ near the fusion boundary.

differed. The fracture appearance in the HAZ exhibited mixed intergranular/transgranular modes, while a completely transgranular fracture mode was observed in the heat-unaffected base metal. Apparently, the rapid heating/cooling cycle during laser welding did not cause any structure changes or cracks in the HAZ of fine-grained Ni_3Al welds, but resulted only in the redistribution of boron at the grain boundaries. For the coarse-grained welds, however, the presence of the HAZ cracks caused the intergranular fracture in that region. Previous studies[11,12] have shown that temperature might contribute to the fractographic transition from ductile into brittle features in the compound. In fact, the results of this study demonstrate that the welding thermal history and the PWHT procedure strongly affect the HAZ fractography of Ni_3Al welds.

The correlation between the amount of boron segregation at Ni_3Al grain boundaries and temperature is in compliance with the Langmuir–McLean equation; i.e., the higher the temperature, the less boron segregated. It is believed that boron desegregation away from grain boundaries might occur during the rapid heating stage of laser welding. Because of the low heat input and rapid cooling rate of the process, boron did not have sufficient time to segregate back to the grain boundaries. As a result, the fracture surface that originally exhibited a transgranular fracture mode in the base metal changed to mixed intergranular/transgranular fracture modes in the HAZ after laser welding. Santella and David[7] suggested that HAZ cracking could be attributed mainly to the interaction between thermal stress and grain boundary weakness at high temperatures. Boron desegregation at the heating stage of laser welding could be used to explain the grain boundary weakness of Ni_3Al at elevated temperatures. However, cracks in the HAZ were not observed for the fine-grained welds. It was suggested that the finer the grains, the lower the stress concentration

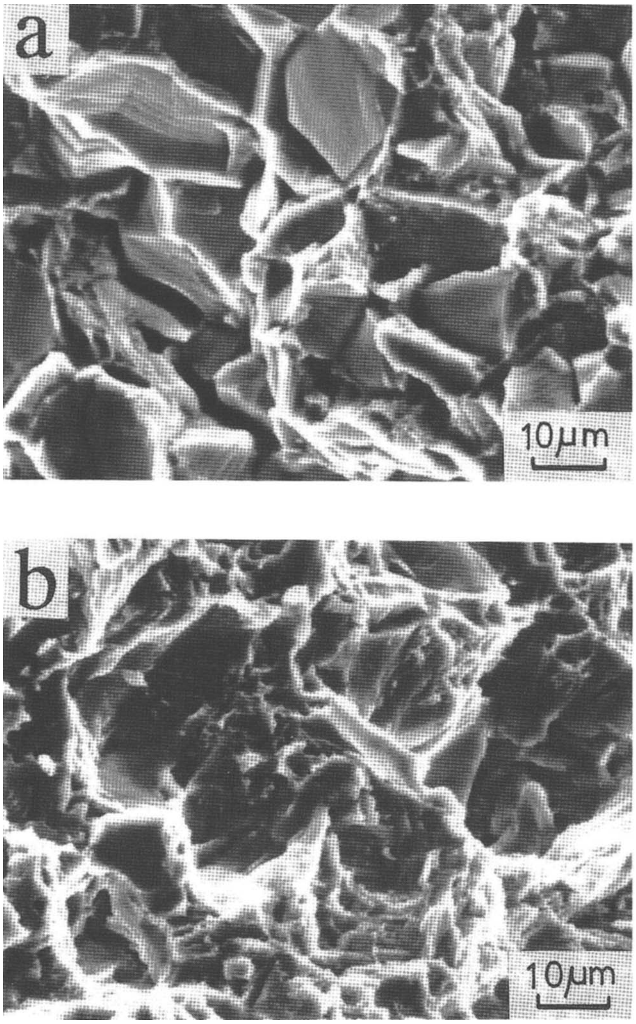

FIG. 9. HAZ fractographs of the C1-16 weld in (a) the as-welded condition and (b) after PWHT at 1000 °C/15 min followed by furnace cooling.

factor in the vicinity of the grain boundary and the higher the applied stress necessary to nucleate a crack.[15]

It was also reported that the grain boundary of Ni_3Al would maintain a lower boron level at elevated temperatures after rapid cooling from 1050 °C, whereas boron would segregate back to grain boundaries in a slow cooling process.[11] The fractographic transition from mixed intergranular/transgranular modes into a completely transgranular mode in the HAZ of fine-grained welds after short-term PWHTs (1000 °C/15 or 30 min + F.C.) might be attributed to the segregation of boron back to grain boundaries during the slow-cooling process. Short-term PWHT changed the fractographic appearance in the HAZ of fine-grained welds (no HAZ cracks); however, coarse-grained welds having intergranular HAZ cracks were not changed. On the other hand, not only fractographic change but also crack healing could occur in the HAZ of coarse-grained welds with only internal cracks after long-term PWHT. Significant grain growth in the welds during long-term PWHT was inevitable. The healing of cracks in the HAZ was preceded presumably by a sintering process, resulting in the change of the fractographic characteristics. The welds after long-term PWHT displayed a more coarse-grained structure than that of the as-welded condition and possibly retained some microvoids.

It was suggested that, other than brittle intergranular fracture, no evidence of liquid films could be found after examining the fracture surface of Ni_3Al + Fe welds containing intergranular cracks in the HAZ.[8] However, liquid films in the HAZ near the fusion boundary on the B3-26 and B5-100 welds were clearly observed in this study. Two possible mechanisms might contribute to the formation of liquid films along grain boundaries in the HAZ of those welds: (1) low melting products caused by segregation of Zr at grain boundaries[16] and (2) backfill of molten metal from the fusion zone to the pre-existent HAZ cracks. The latter seems to be the predominant mechanism on the formation of liquid films in the HAZ of B3-26 and B5-100 welds. In contrast, the former mechanism is the main cause of grain boundary melting of the B3 series specimens that were heat treated at a temperature higher than ~1300 °C for 15 min. Although only few low melting Ni–Zr compounds were found in the HAZ of the weld, it was believed to be associated

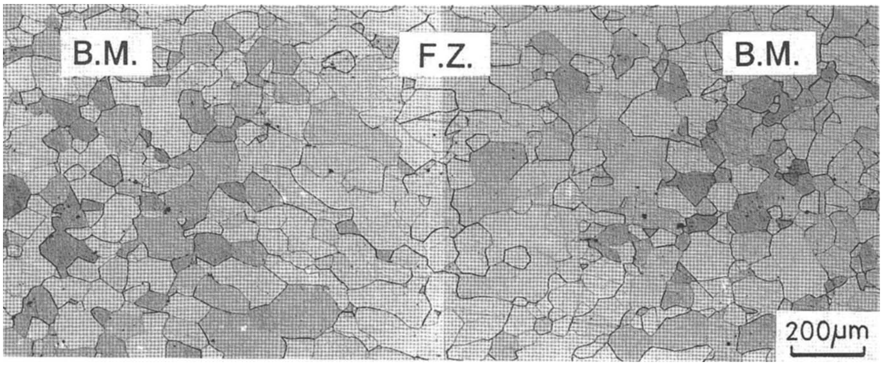

FIG. 10. Optical micrograph of the B3-26 weld after PWHT of 1000 °C/75 h.

with backfilling of molten metal from the fusion zone. Our preliminary studies have confirmed that low melting Ni–Zr compounds coexisted with gamma prime and gamma in the fusion zone of the $Ni_{77}Al_{22}Zr_1$ weld, and they were found mainly in the advancing front of the backfilling liquid.

V. CONCLUSIONS

(1) The welding thermal history and subsequent PWHT strongly affect the HAZ fractography of Ni_3Al welds. Grain boundary weakness resulting from boron desegregation in the heating cycle was responsible for the change in fracture appearance in the HAZ of the weld.

(2) Short-term PWHTs together with slow cooling rates permitted the segregation of boron back to the grain boundaries, resulting in a transgranular fracture mode in the HAZ of impact-fractured welds as compared to that of mixed mode in the as-welded condition. Such a fractographic change was observed only in fine-grained welds; it did not occur in coarse-grained welds containing HAZ cracks.

(3) The formation of liquid films, resulting from backfilling of the molten metal to the cracked grain boundaries, was observed in the HAZ adjacent to the fusion zone of the coarse-grained welds. However, such a phenomenon was not found in the HAZ of the fine-grained welds.

(4) The healing of cracks along with changes in fracture modes and weld structures after long-term PWHT were found in the coarse-grained welds having internal cracks.

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