Microscopic Characterization of Discontinuous Precipitation in a Nickel Aluminide Intermetallic Material

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Abstract

Engineering alloys containing intermetallic compounds such as Ni_3Al have long been known to provide beneficial high-temperature properties including tensile strength, enhanced plasticity and superior resistance to oxidation and creep deformation. Ni_3Al-base alloys may be beneficial as a structural material for high-temperature heat exchangers for nuclear hydrogen generation due its high strength and superior corrosion resistance at elevated temperatures. Heavily cold-worked Ni_3Al can undergo discontinuous precipitation, when subjected to recrystallization annealing. A systematic study of discontinuous precipitation behavior is essential as this phenomenon is expected to cause deterioration in mechanical properties of this otherwise high strength material. In order to study the discontinuous precipitation behavior in Ni_3Al-base alloys, a detailed investigation was pursued on a 70 percent cold-rolled and annealed Ni_3Al(B,Zr) alloy. Well developed cellular precipitates were observed in transmission electron microscopic (TEM) examination of this alloy annealed at 700°C for 2, 5 and 10 hours. The initial stage of discontinuous precipitation was accompanied by thickening of grain boundary along with the formation of semicircular ring-like patterns of these precipitates along the grain boundary. The shapes of these precipitates were found to change to globular or plate-like at higher annealing temperatures. In addition, dissolution (or reversion) of precipitates at high temperatures of annealing was also seen.

Keywords: Nickel aluminide intermetallic compound, cold-work, recrystallization annealing, discontinuous precipitation, precipitation morphology

Work Description:

The beneficial effect of intermetallic compound of nickel aluminide (Ni_3Al) on high temperature tensile properties and resistance to oxidation and creep deformation is well documented in the literature.[1-3] Simultaneously, the detrimental effects of cold-deformation (70 percent) and subsequent annealing at temperatures at and above 700°C are also well-known. An investigation was pursued to characterize the morphology of precipitates in a 70 percent (%) cold-worked Ni_3Al intermetallic compound consisting of nickel (Ni), aluminium (Al), boron (B) and zirconium (Zr). The composition of this alloy is shown in Table 1. The cold-worked material was subsequently subjected to recrystallization annealing at temperatures ranging between 700 and 1000°C for a variable time period.

Table 1

Elements	Nickel	Aluminum	Boron	Zirconium
Atom percent	77.76	21.8	0.1	0.34

The characterization of precipitates by TEM was performed using a Ni_3Al compound subjected to annealing treatment at 700°C and above for durations up to 100 hours. An evaluation of the TEM micrographs of an Ni_3Al, annealed at 700°C, revealed the presence of discontinuous precipitates along the grain boundary, which underwent thickening followed by transformation to ring-like patterns with subsequent conversion to fully developed cellular precipitates. The shape of the cellular precipitates was eventually transformed into globular or plate-like pattern due to the recrystallization annealing performed at higher temperatures. The high temperature annealing of Ni_3Al also resulted in the dissolution of these precipitates, a phenomenon, which is known to render detrimental effect on the performance of alloys containing Ni_3Al type of intermetallic compound.

Results:

- **Optical Microscopy**

The optical micrograph of the test material subjected to homogenization annealing at 1150ºC for 24 hours is shown in Figure 1. This micrograph clearly illustrates the presence of dual-phase (Ni_3Al and NiAl) characterized by γ' and γ, respectively.[4] The γ phase is characterized by dark islands lying in the vicinity of γ' phase. The characterization of both phases by optical microscopy further revealed that the γ' islands containing the secondary γ phase were deformed in the direction of rolling, as illustrated in Figure 2. This deformation of γ phase could be attributed to its relatively greater softness compared to that of γ'. It is interesting to note that this deformation pattern was still retained even after 10 hours of annealing at 700°C, as shown in Figure 3. Some signs of recovery were, however, noted at an annealing temperature of 800ºC, as demonstrated by the recrystallized grains, shown in Figure 4.

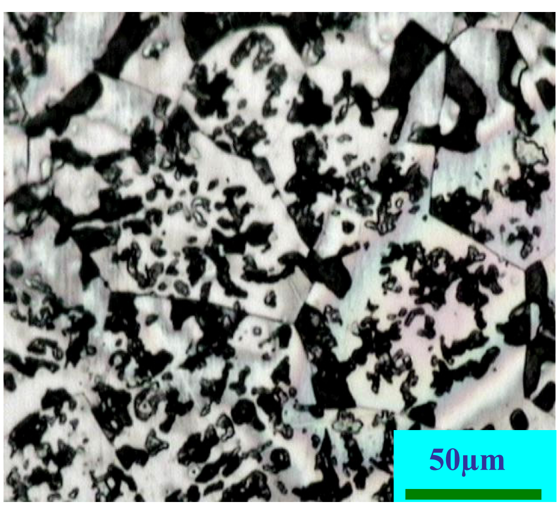

Fig.1 – Optical micrograph of the alloy homogenization annealed at 1150ºC for 24 hours

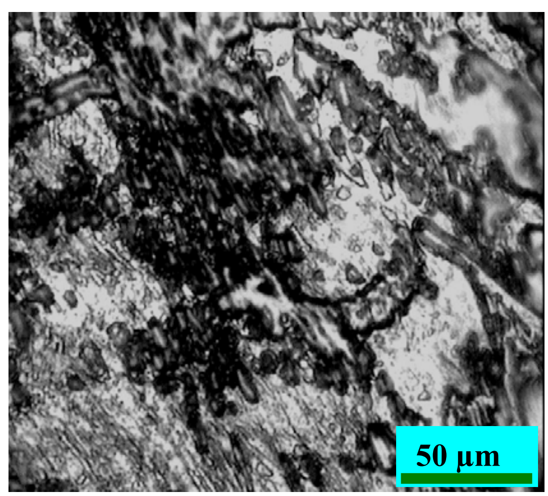

Fig. 2- Optical micrograph after 70 pct. cold reduction

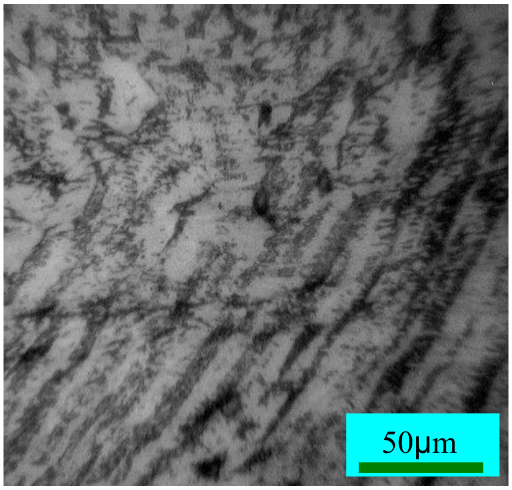

Fig. 3 – Optical micrograph of the sample annealed at 700ºC for 10 hours

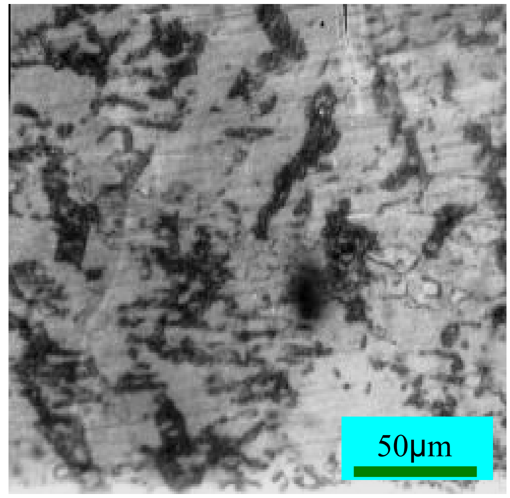

Fig. 4 – Optical micrograph of the sample annealed at 800ºC for 1 hour

Boundary migration, a common phenomenon observed during discontinuous precipitation, was associated with the formation of precipitates behind the migrating grain boundary during annealing at 900ºC for 1 hour, as evident in Figure 5. Annealing at a temperature of 1000ºC eventually caused rapid recrystallization of smaller grains, as shown in Figures 6 through 8. The number of recrystallized grains was, however, enhanced following annealing for longer durations (10 and 100 hours).

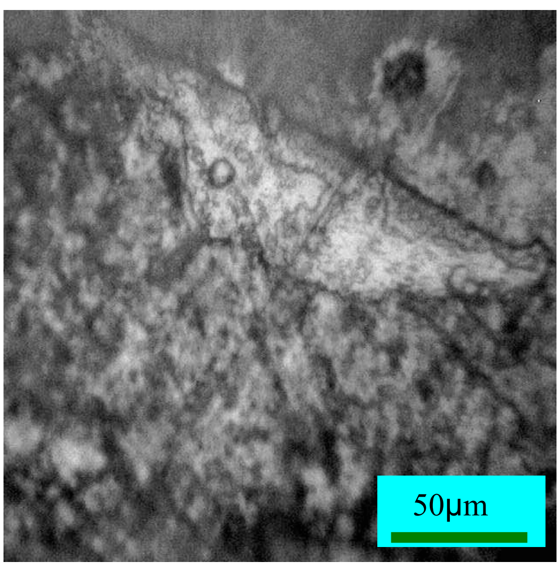

Fig. 5 – Optical micrograph after annealing at 900ºC for 1 hour

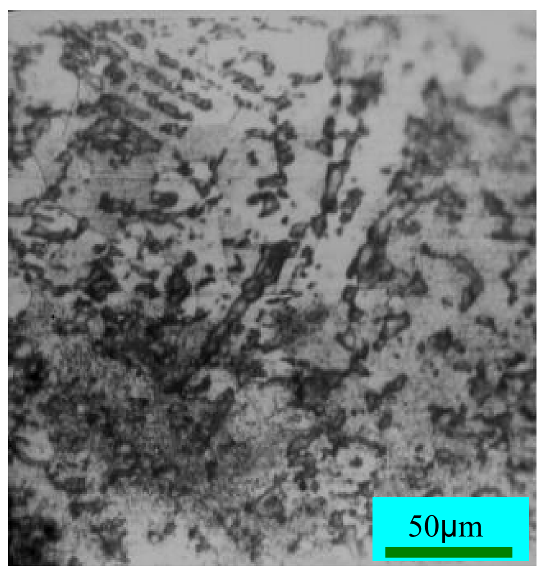

Fig. 6 – Optical micrograph after annealing at 1000ºC for 1 hour

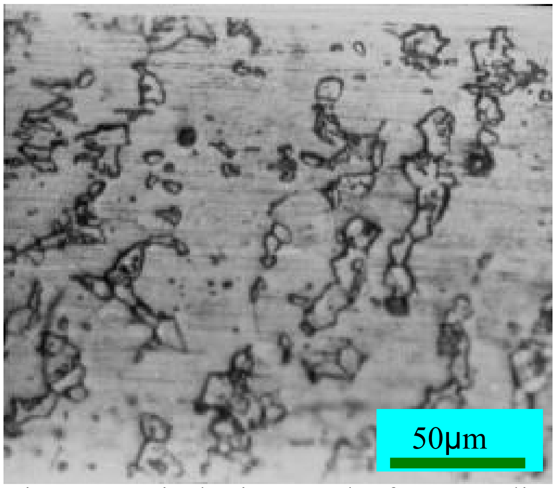

Fig. 7 – Optical micrograph after annealing at 1000ºC for 10 hours

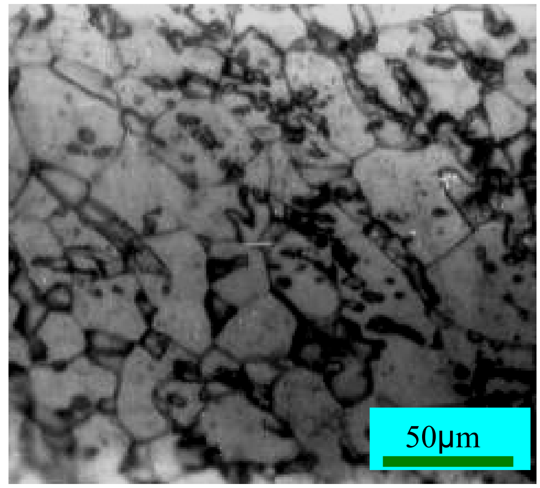

Fig. 8 – Optical micrograph after annealing at 1000ºC for 100 hours

Transmission Electron Microscopy

As indicated earlier in this paper, the characterization of discontinuous precipitates was performed by TEM incorporating strips of test material subjected to different stages of annealing. As illustrated in Figures 9 and 10, the initiation of discontinuous precipitation, was characterized by the thickening of grain boundary due to precipitation followed by the migration of the grain boundary resulting in growth of precipitates. Semicircular ring-like precipitates were formed at the initial stage upon annealing at 700ºC for 1 and 2 hours, respectively. The TEM micrographs shown in Figures 10 and 11, exhibit this phenomenon. The formation of cellular precipitates at a later stage is shown in Figure 12, illustrating the migration of grain boundary front of a recrystallized grain due to annealing at 700ºC for 1 hour.

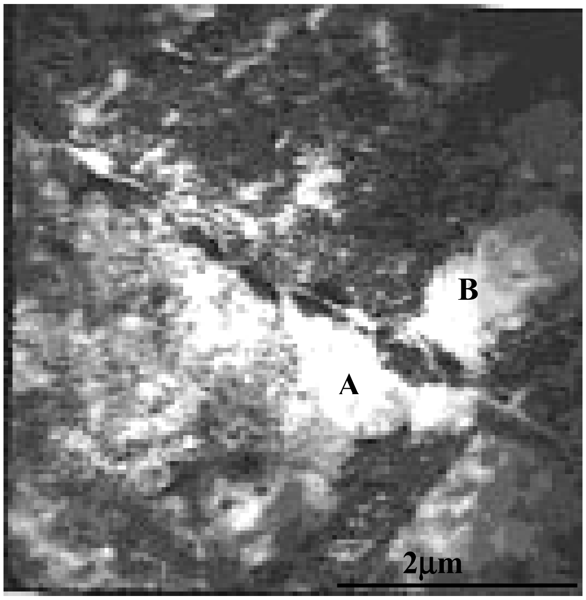

Fig. 9 - TEM micrograph revealing the thickening of grain boundary

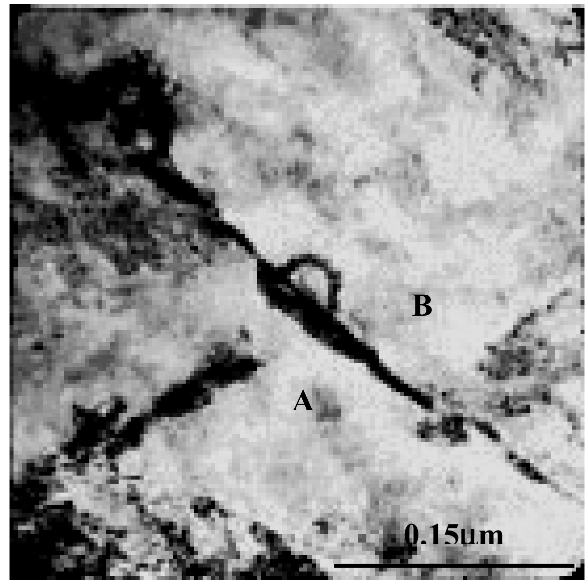

Fig. 10 - TEM micrograph showing the formation of semi-circular ring like feature along the grain boundary

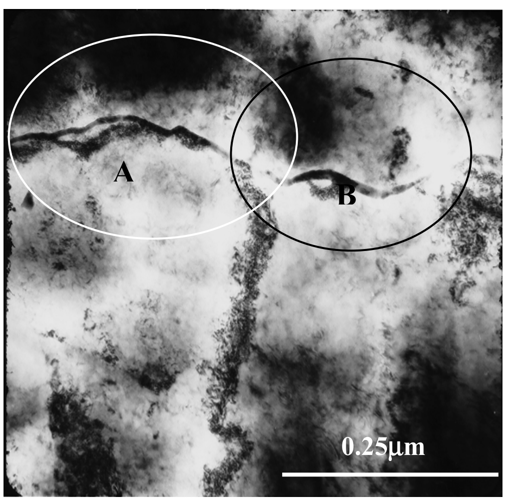

Fig. 11 - Micrograph showing two semi-circular ring like features along with a fully developed precipitate coming down

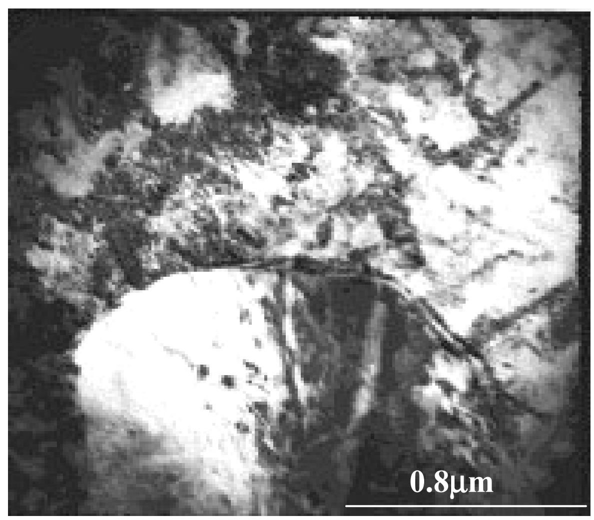

Fig. 12 - TEM micrograph depicting fully developed cellular precipitates behind the migrating grain boundary

It is interesting to note that the precipitates were transformed into plate-like and globular shapes after prolong aging at 700ºC, as illustrated in Figure 13. An area identified by "A" in Figure 13 was further analyzed by selected area diffraction pattern (SADP). Figure 14 illustrates a diffraction pattern showing the absence of any superlattice spot, which is indicative of a recrystallized grain without the characteristics of an ordered γ' phase. This SADP could possibly represent a characteristic of nickel rich disordered γ phase (NiAl).

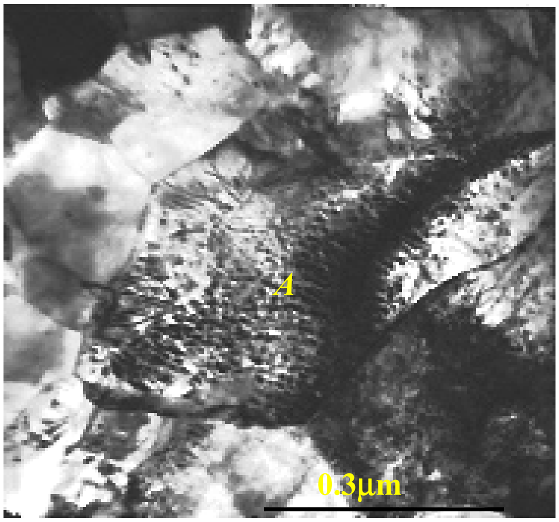

Fig. 13 – Micrograph showing plate-like or globular precipitates after prolong aging at 700ºC

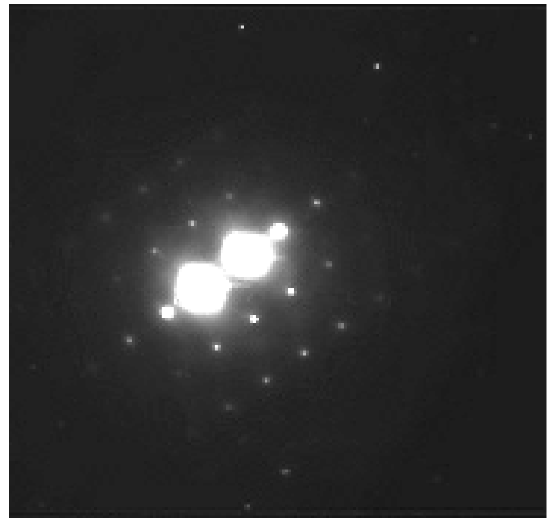

Fig. 14 – SADP showing the absence of any superlattice spot in the region "A" of Fig. 13

A TEM study performed on both sides of the grain boundary of an annealed sample (700ºC for 5 hours) revealed particles showing thin needle or plate like morphology, as shown in Figure 15. The SADP taken from either side (A and B respectively) showed the presence of both matrix and the precipitates, as illustrated in Figures 16 and 17. The distinct streaking of some of the spots indicates the presence of this needle or plate like precipitate particles.

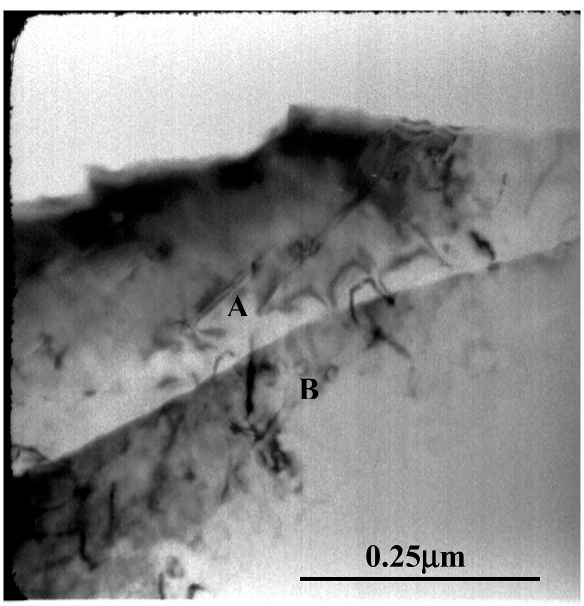

Fig. 15 – Micrograph showing thin needle or plate like morphology of precipitates

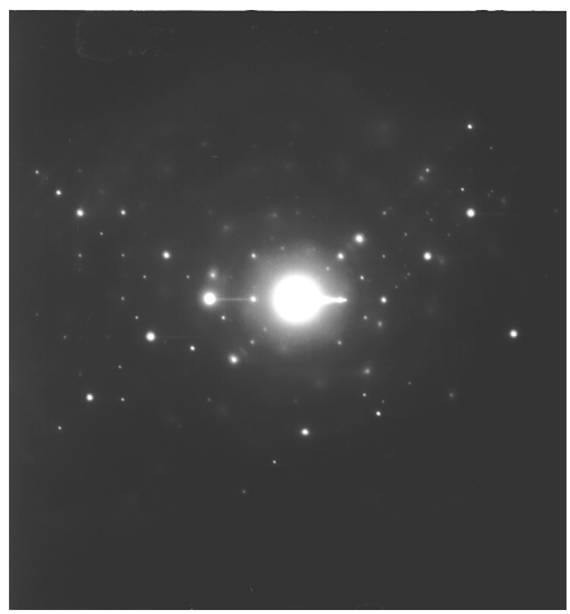

Fig. 16 – Distinct streaking of spots in the SADP taken from region A of Fig. 15, supports thin needle or plate-like morphology of precipitates

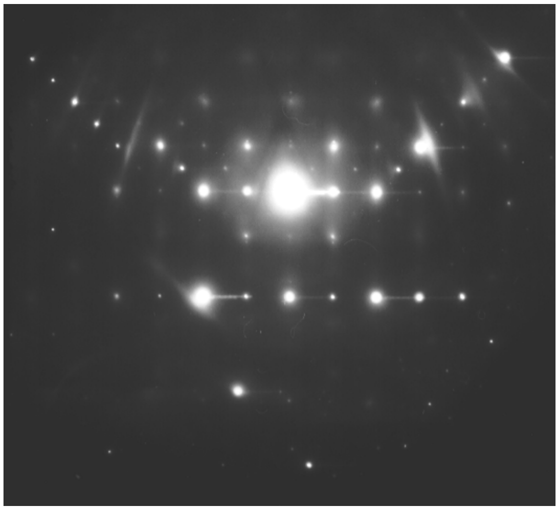

Fig. 17 – SADP taken from region A Fig. 15

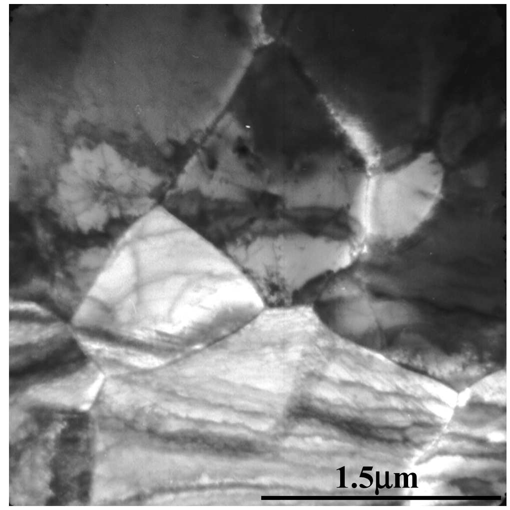

Fig. 18 – TEM Micrograph showing absence of cellular precipitates

Annealing at temperatures above 700°C for comparable durations resulted in dissolution of precipitates in the matrix. A series of micrographs (Fig. 18 to Fig. 20) show the general microstructures obtained at the annealing temperature of 900°C, which clearly indicate the absence of discontinuous precipitation or sometimes, the presence of a, few fine precipitates as shown in Figure 21.

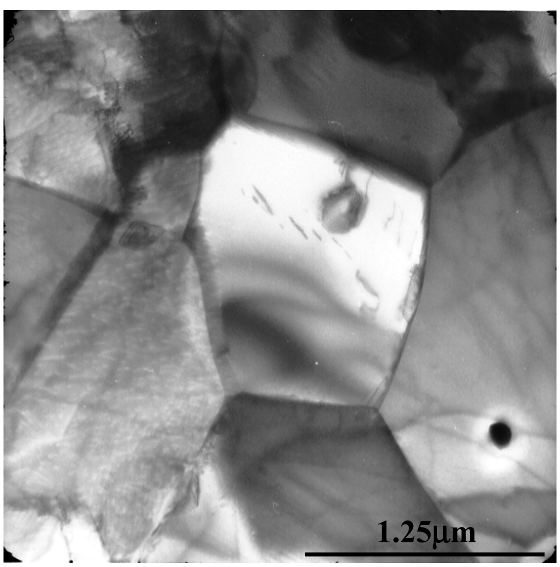

Fig. 19 – Micrograph showing very fine precipitates within the central grain

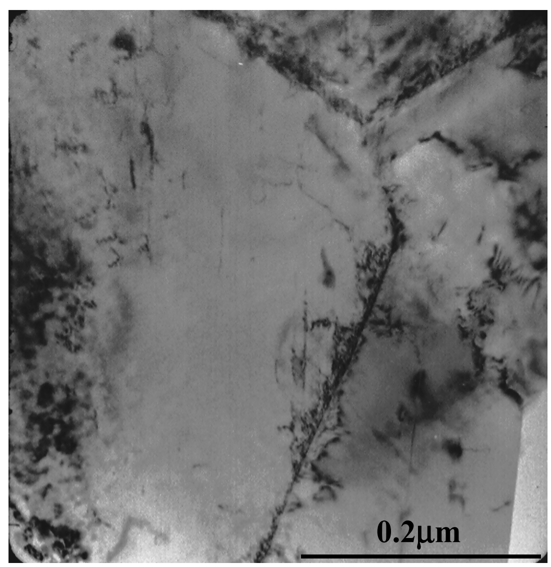

Fig. 20 – TEM micrograph showing very fine precipitates along the grain boundary

Results

The objective to carry out a detailed study of the discontinuous precipitation phenomenon during recrystallization annealing was successfully achieved. Discontinuous precipitation in Ni_3Al(B, Zr) alloy was found to start at 700°C, with thickening of the boundary and formation of a semi-circular ring like feature along the boundary in the initial stage, followed by formation of fully developed cellular precipitates at a later stage. At higher temperatures of 900°C and 1000°C, the precipitates were found to dissolve back into the matrix.

References

1. Dongyun Lee, M.L. Santella, I.M. Anderson, and G.M. Pharr; *Metallurgical and Materials Transactions A*, *Volume 36A, Issue 7, July 2005, p.1855*.

2. Satoru Kobayashi, Masahiko Demura, Kyosuke Kishida and Toshiyuki Hirano; *Intermetallics, Volume 13, Issue 6, June 2005,* p. *608*.

3. Takeshi Izumi, Takumi Nishimoto and Toshio Narita; *Intermetallics, Volume 13, Issue 7, July 2005,* p.727.

4. B. Bhattacharya and R. K. Ray; *Metallurgical and Materials Transactions A*, *Volume 31 A, December 2000*, p. 3011.