

Recovery and recrystallization processes in binary Fe₃Al based alloys

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Three binary alloys of composition Fe-24Al, Fe-28Al and Fe-34Al were prepared to study the recovery, recrystallization and grain growth processes in Fe₃Al based alloys. These alloys were rolled and annealed at temperatures in the range 873 K to 1273 K for two hours. Grain size measurements were performed as a function of composition, annealing temperature and time. Transmission electron microscopy showed recovered and recrystallized grains after annealing at 873 K. The mechanisms of recovery processes was found to be by the migration of single dislocations towards each other to form linear arrays which can subsequently form square or hexagonal dislocation networks. Recrystallization can take place either by the enclosure of dislocation free regions by dislocation networks or by the preferential growth of subgrains. The composition dependence of the recovery and recrystallization processes is weak. © 2000 Kluwer Academic Publishers

1. Introduction

Intermetallic compounds are being intensively studied for high temperature structural applications. Fe₃Al based alloys are one such class of compounds which are potential replacements for stainless steels and some nickel base superalloys [1, 2]. These low cost alloys possess good strength and excellent oxidation and sulphidation resistance. Applications include engine compressor blade and housings, heat exchangers, furnace fixtures as well as piping and tubing for power generation and chemical industries [3]. The crystal structure of these alloys in the region of interest is b.c.c. at high temperatures, B2 at intermediate temperatures and D0₃ at low temperatures (Fig. 1). Two factors limit the further use of these alloys: their poor room temperature ductility and low strength above 873 K [4]. Several approaches have been developed to overcome these limitations including additions of alloying elements such as chromium and a combination of themomechanical and heat treatments designed to produce metastable B2 at room temperature [5].

Recovery and recrystallization has been studied in these alloys primarily because of (a) the increased capacity to hot work these materials through strain relief, (b) to control the grain size and texture and (c) improve the mechanical properties of these alloys [4–8]. Sluggish dislocation recovery has been suggested to limit recrystallization by slowing the grain nucleation rate in Fe₃Al [9]. Increased dislocation density has also been shown to retard the B2 to D0₃ transformation [10] and provide enhanced ductility and strength [8]. The role of alloying additions on the recrystallization temperature as well the effect of extent of recrystallization on room temperature tensile ductility have been evaluated [11, 12]. Earlier studies also showed that a highly elongated

microstructure, with minimum transverse grain boundaries, is the most resistant to hydrogen diffusion [11]. Increased recrystallization increased the number of transverse boundaries, which increased the hydrogen diffusion and resulted in lower ductility values. Recrystallization can also shift the fracture mode from mainly transgranular to intergranular. A highly elongated unrecrystallized microstructure of a Fe-28.01 Al-5 Cr-0.1 Zr-0.04 B alloy increased the room temperature tensile elongation to 20%. These studies have shown that the tensile ductility and strength values are a sensitive function of the extent of recrystallization and the extent of recrystallization can be observed by optical metallography in the temperature range of 873 to 1073 K.

In addition to these practical considerations, there is considerable interest in the question of how recovery, recrystallization and grain growth (RRG) processes are changed by annealing in the vicinity of the order/disorder temperature. Several workers have found in other ordered alloys that such annealing does play a role in changing the RRG processes. In L1₂ compounds (e.g., Cu₃Au and (Co, Fe)₃V), grain boundary mobility is strongly retarded by ordering. A complex interplay between order and recrystallization kinetics is also often observed [13]. Interestingly, FeCo based compounds also show increased ductility if the microstructure was partially recrystallized. Compared to the L1₂ case, much less information is available on B2 based compounds. Studies on NiAl showed that recrystallization and grain growth kinetics were of the normal form. The studies of Jiraskova *et al.* [14], Morris [15] and Morris and Gunther [9] have related the degree of order to the mechanisms of RRG processes, particularly in the vicinity of the D0₃/B2 transition. However, aspects related to the role of alloy composition and the

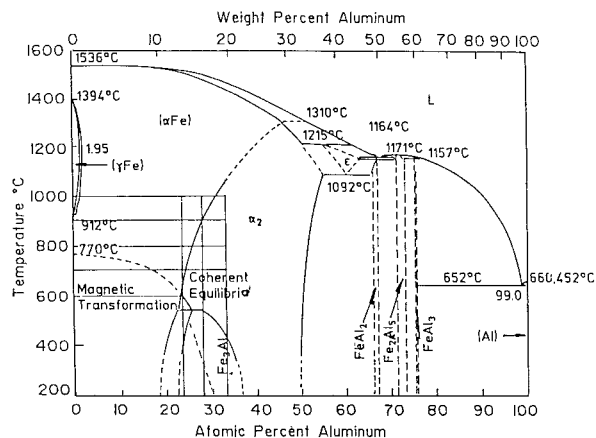


Figure 1 Phase diagram of Fe-Al with relevant compositions and annealing temperatures marked.

mechanisms in the vicinity of the B2/b.c.c. temperature have received less attention.

The earlier studies mentioned above have already identified the temperature range in which RRG processes occur, mainly by optical and scanning electron microscopy, hardness and resistivity. They have also studied superdislocation splitting and order parameter changes. These studies provided the base for the present investigation, which focussed on grain size measurements and transmission electron microscopy studies. Since RRG processes consist of several heterogeneous and statistical processes, it is necessary to study these processes as a function of temperature *and* composition. Many micrographs are required to obtain a consistent model of the RRG processes, which will explain the sequence of microstructural evolution. The samples can be highly magnetic and therefore only a few detailed TEM investigations have been performed [9]. Hence, to obtain greater insight into the mechanisms of RRG processes in Fe₃Al alloys as a function of temperature and alloy composition, three binary alloys of composition Fe-24Al, Fe-28Al and Fe-34Al (atomic percent) were rolled and annealed at various temperatures selected on the basis of existing literature. Foils prepared from these samples were examined by transmission electron microscopy (TEM) and these results are reported in this paper.

2. Experimental procedure

Three alloys of nominal composition Fe-24Al, Fe-28Al and Fe-34Al weighing 0.4 kg each were prepared by arc

TABLE I Average grain size (in microns) for Fe-24Al, Fe-28Al and Fe-34Al annealed for 2 h. between 873 K and 1273 K

Annealing temperature (K)	Fe-24Al	Fe-28Al	Fe-34Al
As-rolled	**	**	**
873	**	**	**
973	60	78	67
1073	113	91	85
1173	114	137	114
1273	146	148	191

**Quantitative measurement on highly pancaked structures were not concluded.

melting and drop casting into 12.7 × 25.4 × 127 mm bars. These bars were then hot forged 25% at 1273 K, stress relieved at the forging temperature and hot forged again 33% to a final thickness of 6.4 mm on a 500 ton Baldwin forging press. After forging, the alloys were hot rolled 60% to a thickness of 2.5 mm on a Bliss 4 high rolling mill with a roller surface speed of 132 mm/s at approx. 10%/pass. Finally, the alloys were stress relieved at 923 K for 10 min and warm rolled 70% at 923 K to a final thickness of 0.8 mm under similar rolling conditions. Samples from these rolled strips were heat treated in the temperature range of 873 to 1273 K for 2 h, followed by air cooling. TEM foils were prepared in

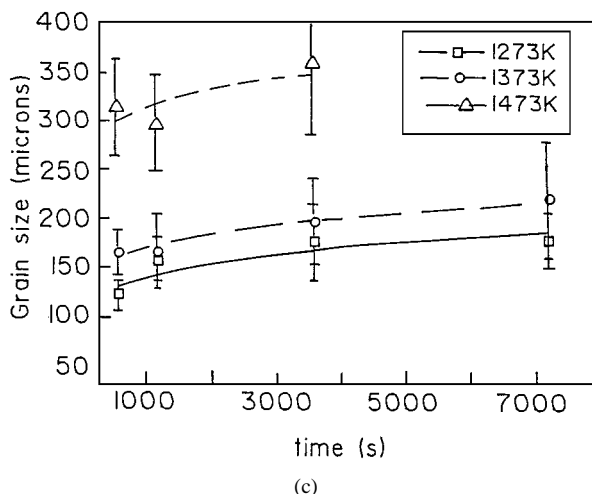
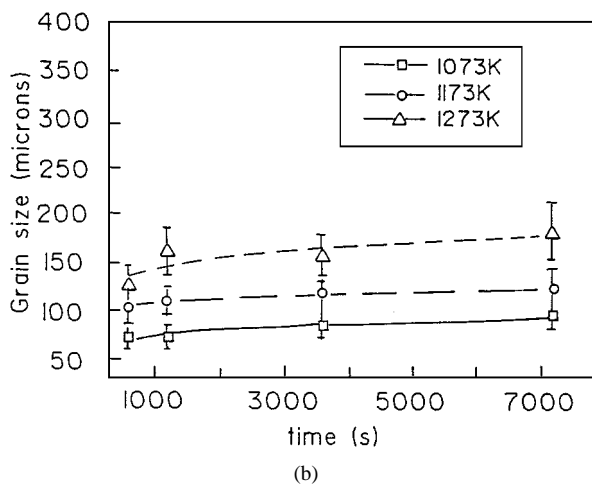
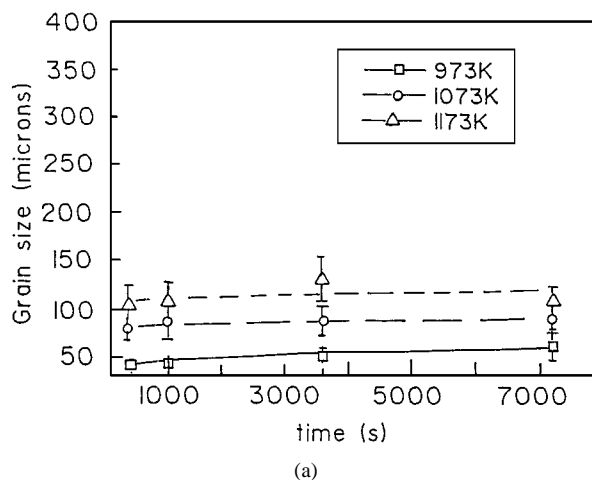
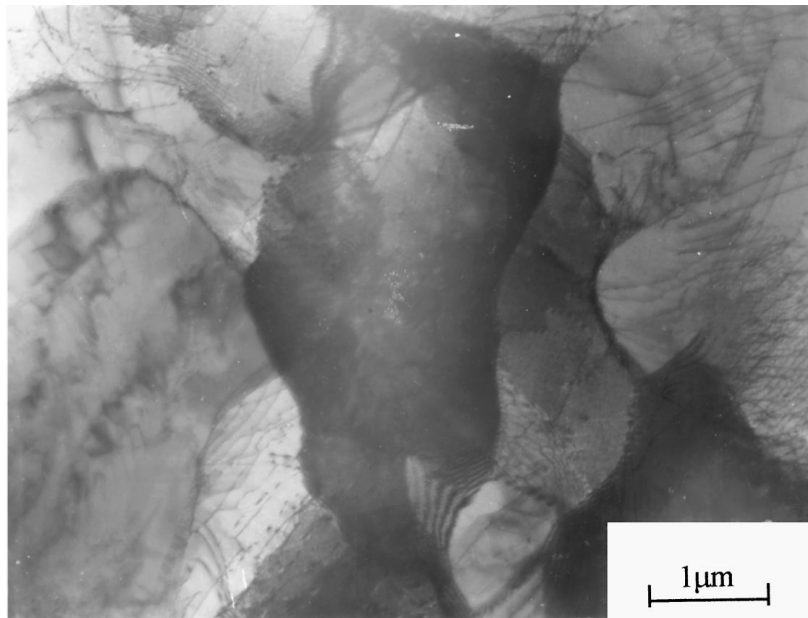


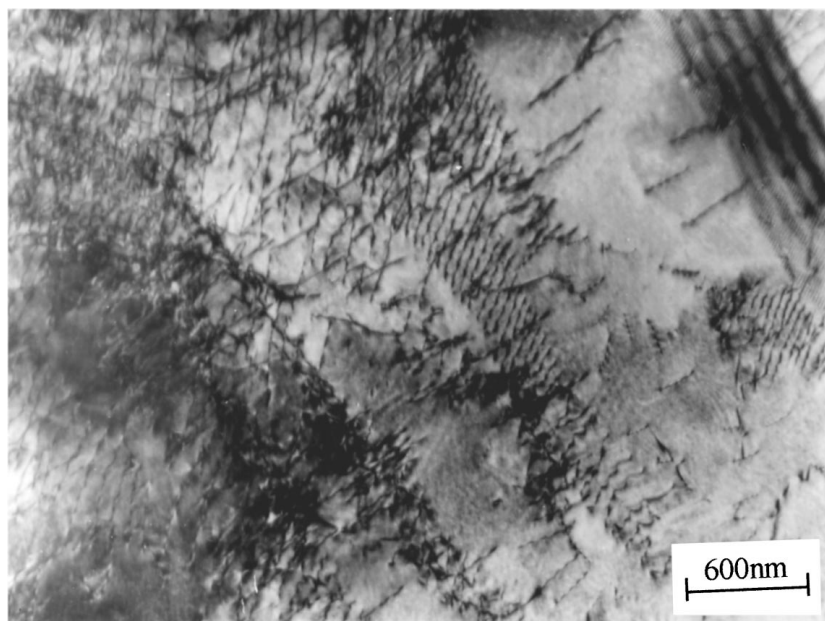
Figure 2 Average grain size versus time for (a) Fe-24Al, (b) Fe-28Al and (c) Fe-34 Al alloys.

TABLE II Grain size (in microns) for Fe-24Al, Fe-28Al and Fe-34Al annealed for various times between 873 K and 1273 K

Alloy	Time (s)	973	1073	1173	1273	1373	1473
Fe-24Al	600	42 ± 7.7	78 ± 15.5	104 ± 20.7			
	1200	44 ± 7.4	85 ± 19	107 ± 19.3			
	3600	51 ± 8.3	86 ± 17.2	129 ± 23			
	7200	59 ± 14.4	87 ± 10.7	107 ± 12.1			
Fe-28Al	600		73 ± 14.6	103 ± 16.8	129 ± 17.2		
	1200		71 ± 13.7	109 ± 15.8	160 ± 25.5		
	3600		85 ± 14.9	118 ± 26.9	156 ± 22.3		
	7200		93 ± 14.7	123 ± 20	181 ± 30		
Fe-34Al	600				121 ± 16	165 ± 23.5	313 ± 49.7
	1200				156 ± 22.3	165 ± 37.8	294 ± 50.8
	3600				174 ± 39.2	195 ± 43.6	357 ± 74.7
	7200				174 ± 28.2	216 ± 59.3	

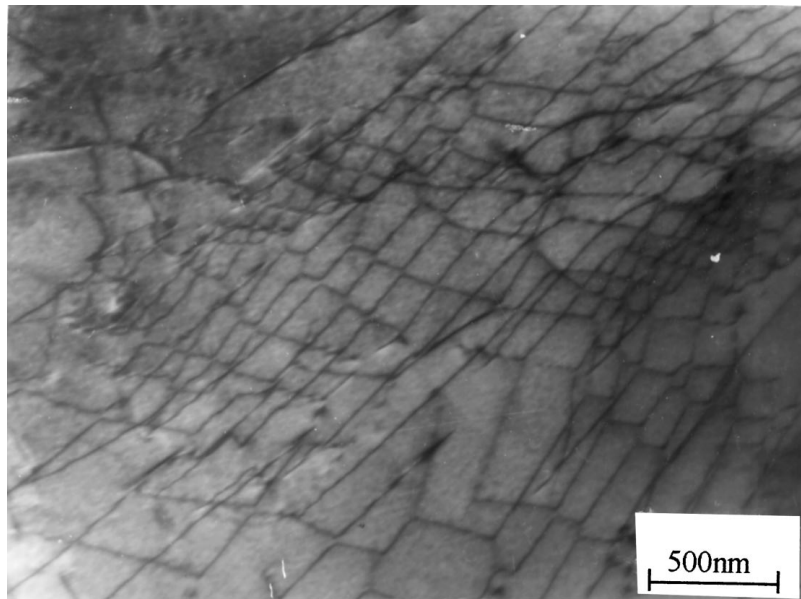


(a)

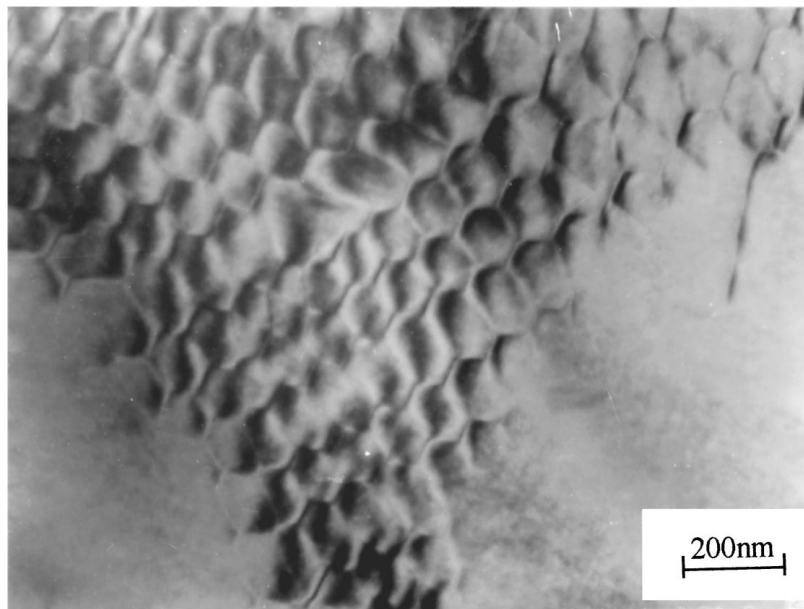


(b)

Figure 3 BF TEM micrograph of Fe-24Al annealed at 873 K, (a) showing elongated grains of average size 2 μm, (b) linear arrays of dislocations (c) rectangular dislocation networks and the stepped nature of single dislocations, (d) square and hexagonal dislocation networks. (Continued)



(c)



(d)

Figure 3 (Continued).

a twin jet polishing system using a 3 : 1 methanol : nitric acid electrolyte solution, operating at 14 V and 200 mA at 293 K. Grain size measurements were made on cross sectioned alloys polished and etched with a solution of 5 ml water, 25 ml acetic acid, 15 ml HCl and 15 ml HNO₃. Grain size measurements were determined using the linear intercept method and averaged over 10 measurements.

3. Results

The grain size of the Fe-24Al, Fe-28Al and Fe-34Al alloys as a function of temperature are tabulated in Table I. The as-rolled condition was a highly pancaked structure in all 3 alloys, with grain size ranging from 10 μm width and 40 to 450 μm length. This structure was retained in all 3 alloys heat treated upto 873 K. At 973 K, the average grain size was 60, 78 and 67 μm for Fe-24Al, Fe-28Al and Fe-34Al alloys, respectively.

At higher temperatures, average grain size increases upto 191 μm at 1273 K. The Fe-34Al alloy showed a marked increase in grain size between 1173 K and 1273 K, which is just above the α/B2 order-disorder temperature.

The kinetics of grain growth was studied by measuring grain size at various times for selected temperatures. These temperatures were selected on the based on the α/B2 order-disorder temperature for each composition. Grain size was measured after 600, 1200, 3600 and 7200 s (Table II). Fig. 2a–c show the relationship between grain size and time for each temperature and composition. Grain growth follows the relation:

$$D = D_0 + A\gamma t^n$$

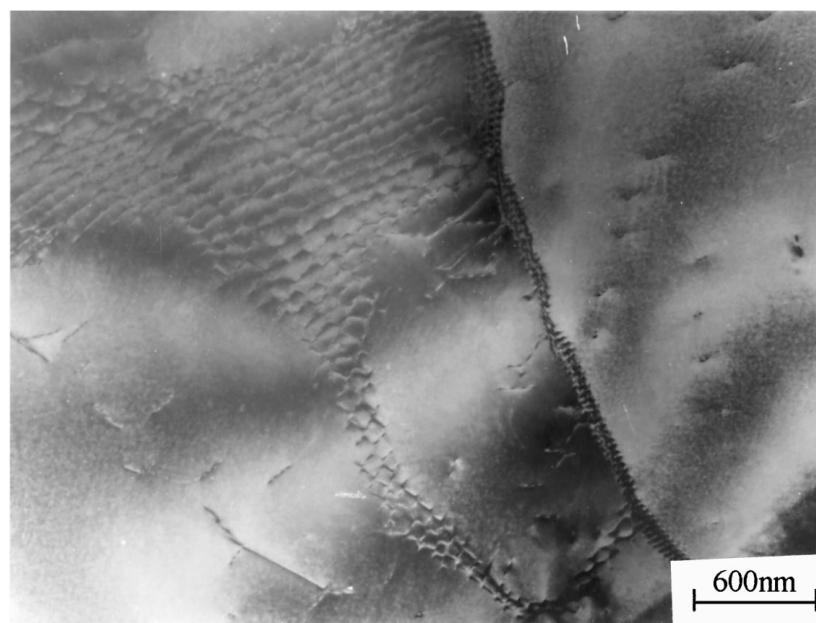
where n is ideally 0.5. Since the starting materials was as-deformed, the final grain size is a function of RRG

kinetics. Quantitative analysis of grain growth exponents cannot be obtained from these curves since the incubation time for recrystallization at these temperatures is less than 600 s. Also, due to sample size constraints the grain size cannot exceed 500 μm without intersecting a surface. The grain size after 600 s does not vary much with composition. For example, at 1073 K, the grain size of Fe-24Al (for which the $\alpha/\text{B2}$ order-disorder temperature is 973 K) and Fe-28Al (for which $\alpha/\text{B2}$ order-disorder temperature is 1173 K) was 77 and 72 μm , respectively. Therefore, the ordering temperature had only a weak effect on the kinetics.

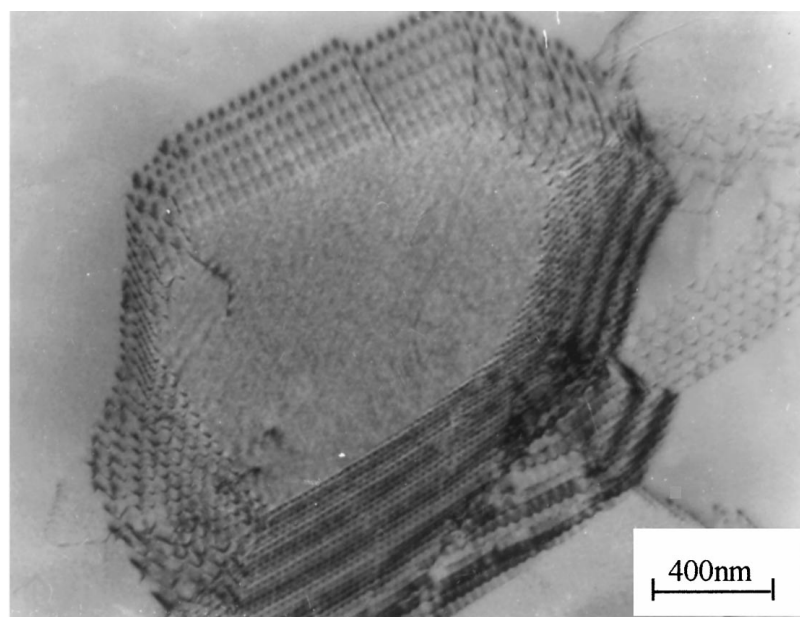
In the Fe-24Al alloy, after annealing at 873 K in the $\alpha + \text{B2}$ phase field, a heterogeneous microstructure was produced, with different regions showing different

extents of recovery and recrystallization. Many grains of average size 2 μm were observed, often containing long straight dislocations (Fig. 3a). Various stages of recovery could be inferred: Linear arrays of dislocations which formed low angle tilt boundaries were observed and the mutual interaction of dislocations in these arrays can lead to the formation of irregular networks (polygonization) through bending to form networks of dislocations (Fig. 3b). Hexagonal and square dislocation networks, with an average spacing of 100 nm between parallel dislocations, were seen (Fig. 3c and d).

Subgrains formed by these dislocation networks were observed (Fig. 4a and b). Triple junctions formed by the intersection of both square and hexagonal networks were commonly seen and the average spacing

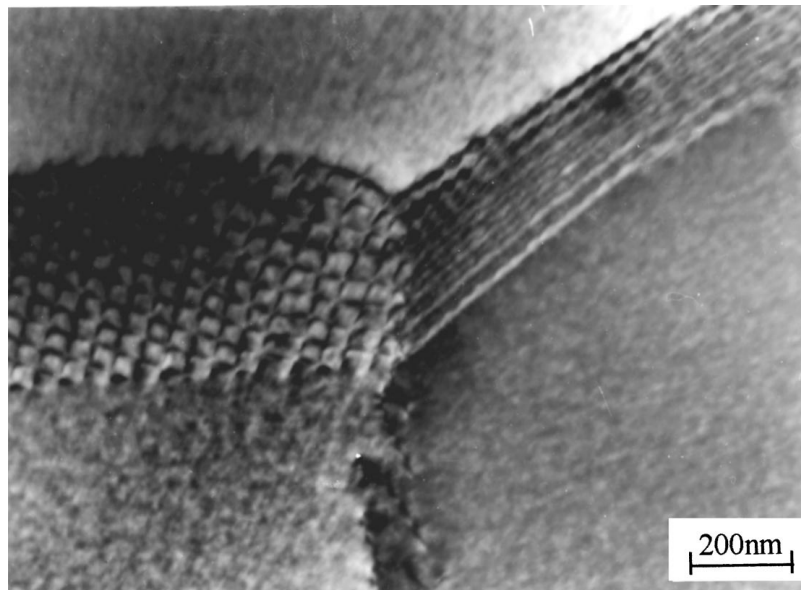


(a)

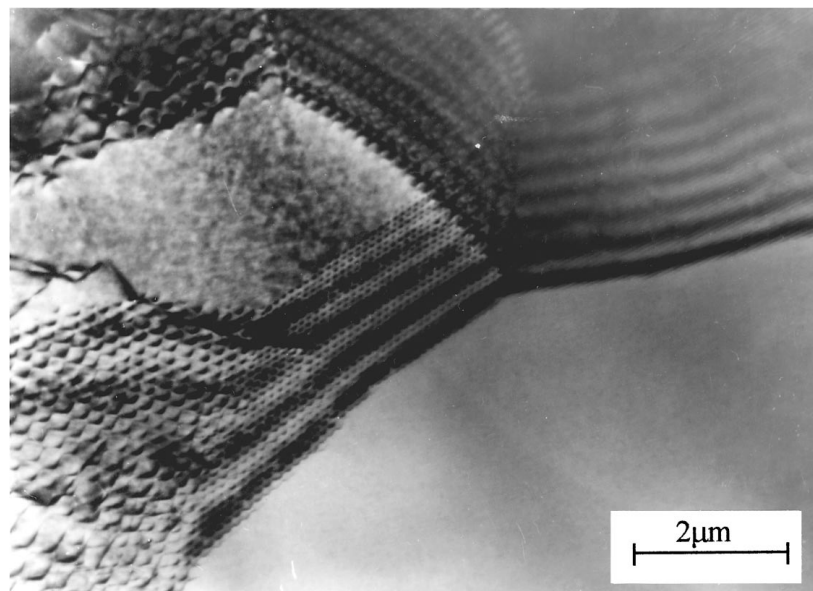


(b)

Figure 4 BF TEM micrograph of Fe-24Al annealed at 873 K (a) subgrain formation associated with dislocation networks, (b) subgrains enclosed by dislocation networks, (c) triple junctions formed by the intersection of well formed dislocation networks (d) a triple junction and the associated dislocation network. (Continued)



(c)



(d)

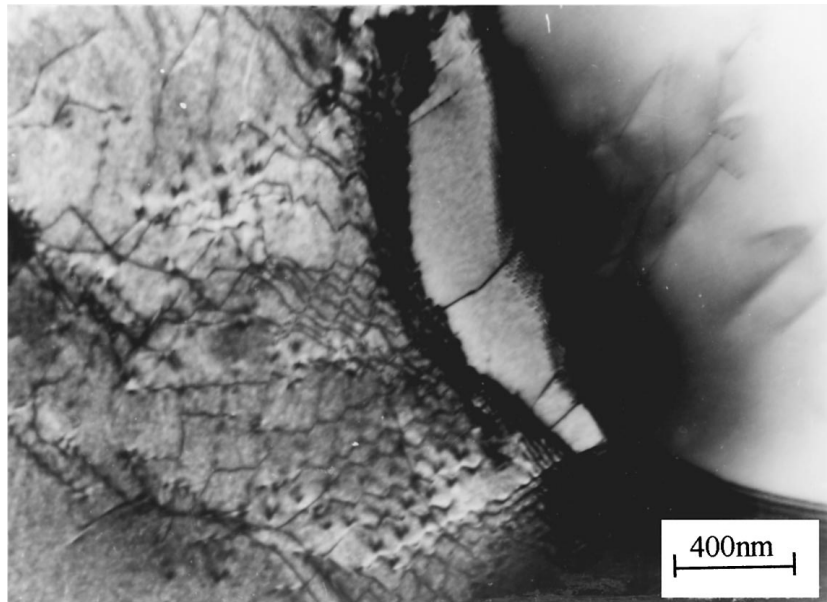
Figure 4 (Continued).

between parallel dislocations in these well formed networks was 30 nm (Fig. 4c and d). Recrystallized grains (average size $1 \mu\text{m}$) were also observed; one side of the grain boundary of such grains was essentially free of dislocations, while on the other side polygonized dislocation networks were observed (Fig. 5a). Interestingly, the migration of high angle grain boundaries parallel to themselves could be inferred from Fig. 5b, since displacement fringes have formed at the position of the grain boundary prior to migration. The migrating boundaries have long trailing dislocations in a few areas. Displacement fringes were also observed in some grains and these features have been attributed to the condensation of impurity atoms on the (100) planes (Fig. 5c) [9]. In the samples annealed at 973 K in the vicinity of the B2/b.c.c. order-disorder transition temperature, and at higher temperatures in the b.c.c. phase

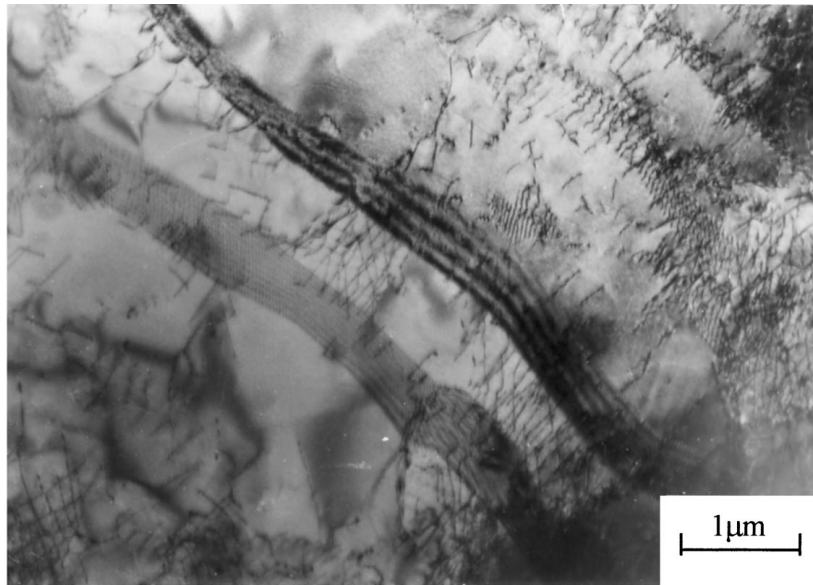
field, recrystallization and grain growth was advanced and large equiaxed grains with high angle grain boundaries were observed.

The Fe-28Al alloy in the as-rolled condition displayed a large number of dislocation tangles as well as irregular arrays of parallel dislocations (Fig. 6a and b). Annealing at 873 K in the B2 phase field showed that several subgrains of approx. $1 \mu\text{m}$ size had formed, with a substantial reduction in the dislocation density, and such subgrains were separated by low angle boundaries with small relative misorientation (Fig. 6c and d).

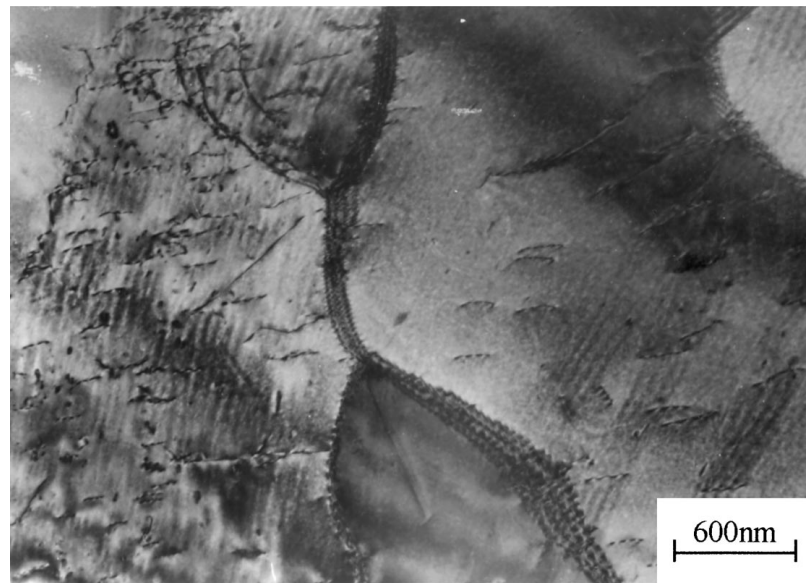
As in the case of the Fe-24Al alloy, linear, square and hexagonal networks of dislocations were observed (Fig. 7a and b). Recrystallized grains were also observed and the matrix around such grains contained a high density of dislocations (Fig. 7c and d). Occasionally, large strain-free grains (approx. $8 \mu\text{m}$ in size)



(a)

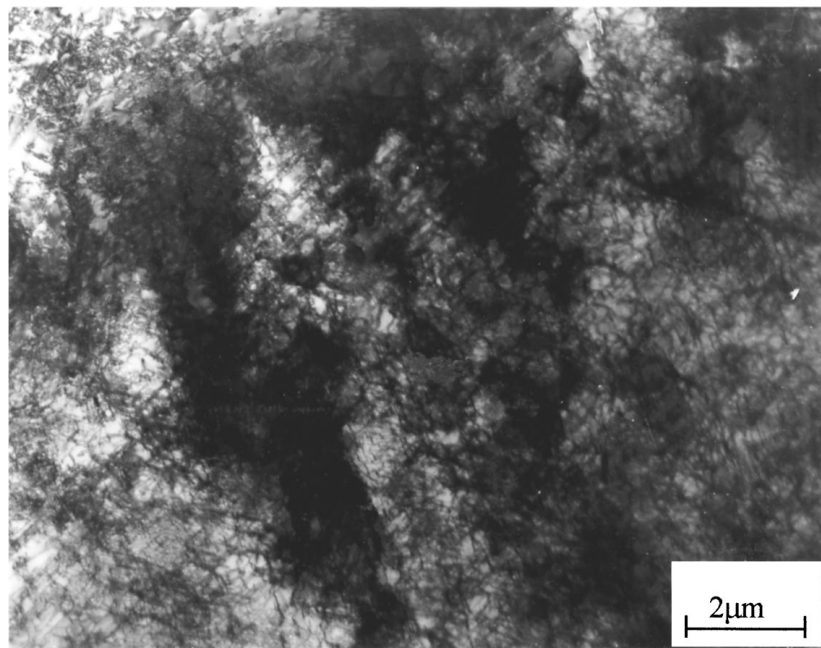


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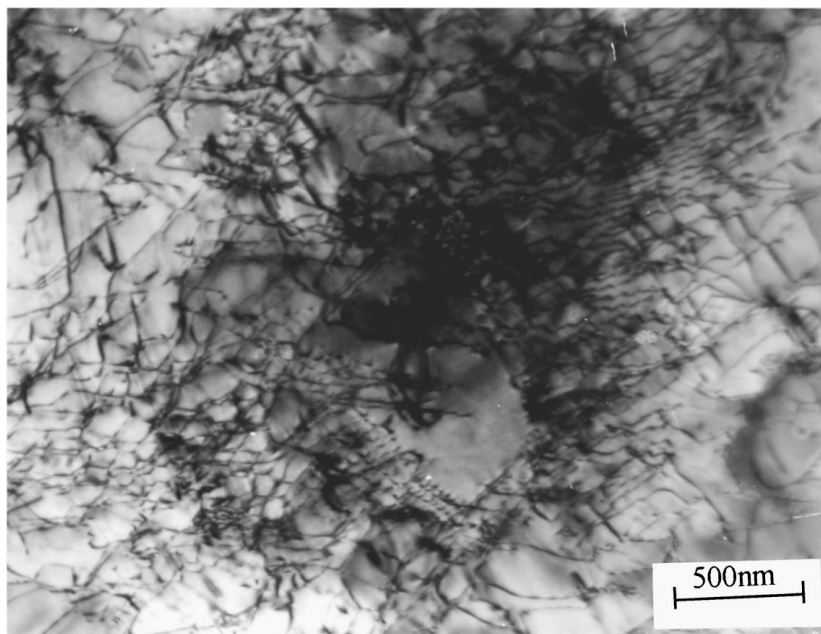


(c)

Figure 5 BF TEM micrograph of Fe-24Al annealed at 873 K (a) recrystallized grains can be observed in a grain containing single dislocations and a loose dislocation network, (b) a grain boundary with trailing single dislocations, (c) triple junctions and displacement fringes observed within large grains.



(a)



(b)

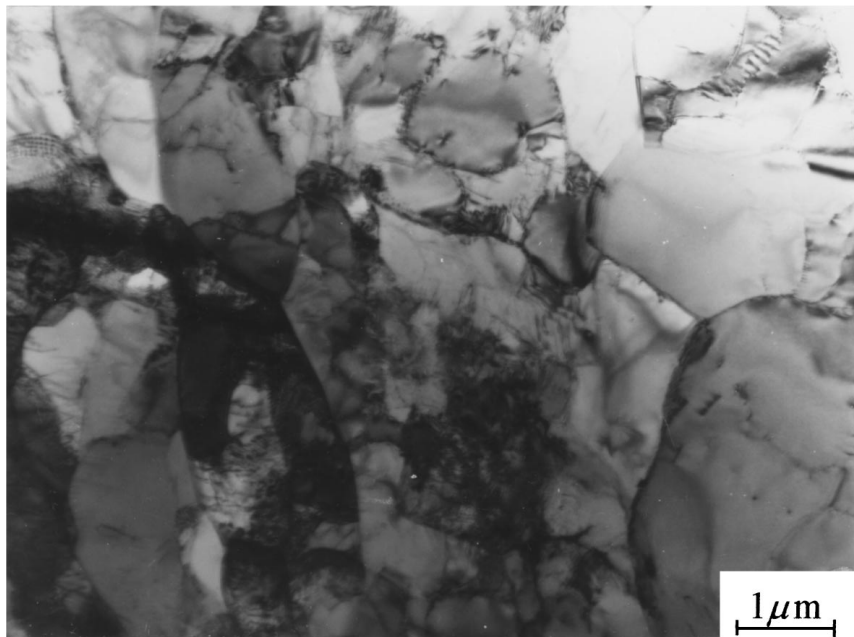
Figure 6 BF TEM micrograph of Fe-28Al in the as-rolled condition (a) revealing a large density of dislocations, (b) single dislocations and loose dislocation network formation, (c) BF TEM micrograph of Fe-28Al annealed at 873 K, in which subgrain formation can be observed, (d) BF TEM micrograph of Fe-28Al annealed at 873 K, revealing low angle dislocation boundaries formed between two subgrains. (*Continued*)

surrounded by small sub grains were observed (Fig. 7e) and at higher temperatures (973 K to 1273 K), recrystallization and grain growth led to the formation of large equiaxed grains (Fig. 7f).

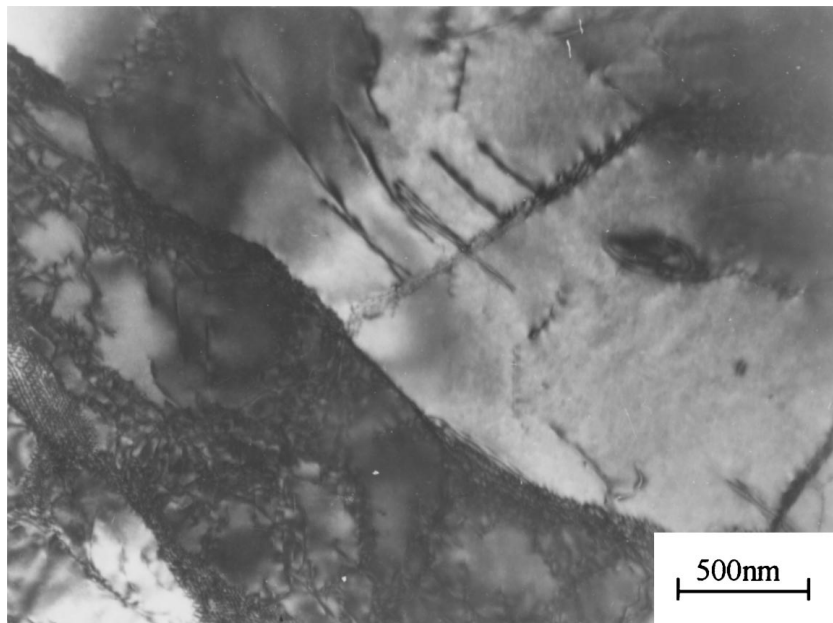
The Fe-34Al alloy in the as-rolled condition showed dislocation tangles, slip bands and in a few areas, an irregular array of dislocations (Fig. 8a–c). Annealing at 873 K in the B2 phase field resulted in the formation of hexagonal dislocation networks as well as parallel single dislocations, with superjogs coexisting with square dislocation networks (Fig. 8d and e). After annealing at 973 K and higher temperatures, large equiaxed grains were observed, corresponding to extensive grain growth (Fig. 8f).

4. Discussion

The above results on Fe-24Al, Fe-28Al and Fe-34Al alloys showed that the RRG processes which occurred on annealing in the temperature range 873 K to 1273 K are highly heterogeneous with various processes occurring simultaneously in various parts of the sample. These processes showed only a weak dependence on Al level. This behavior can be compared with other b.c.c. based materials. The present results are broadly consistent with earlier work [3–6], although direct comparison is not possible due to compositional differences; previous work was primarily conducted on complex alloys. Earlier studies had indicated that RRG processes would depend on composition, initial microstructure, retained



(c)



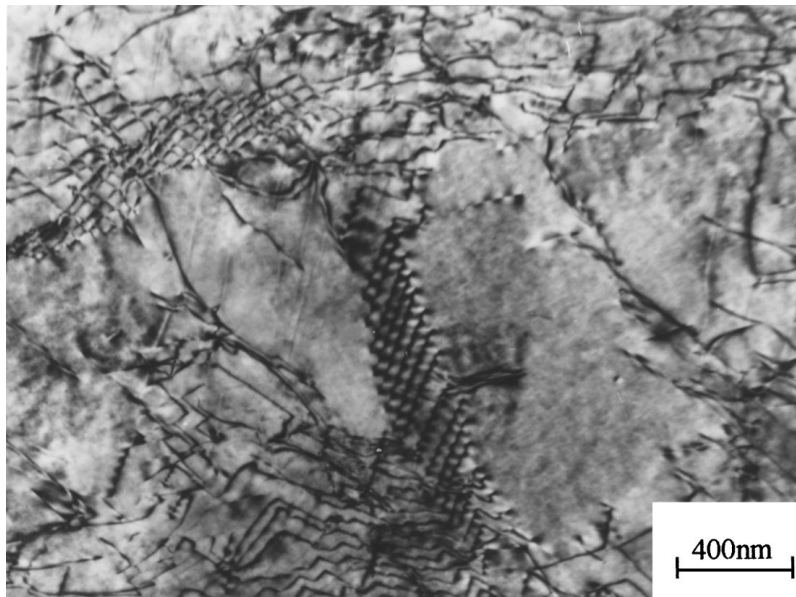
(d)

Figure 6 (Continued).

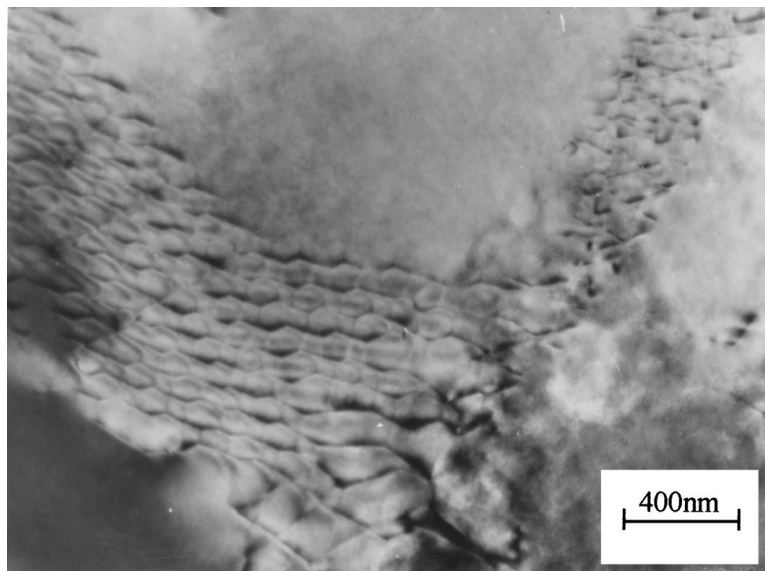
cold work and type and extent of order [8]. In the case of FeAl alloys [16], there are only small differences in recrystallization temperature as Al content is increased from 40 to 48 Al. The results of Jiraskova *et al.* [14] on Fe-28Al indicated that the recovery process consisted, sequentially, of (a) annihilation of defects, dislocation straightening and glide, (b) dislocation cross slip and glide (c) cell wall sharpening, annihilation of cell walls and transformation into subgrains or grains. This last step was connected with the B2/D0₃ ordering transition. Morris and Gunther showed that recrystallization had taken place by simplification of dislocation tangles leading to cell and subgrain formation. Some of these subgrains transformed to new grains. In the vicinity of the B2/D0₃ ordering transition, the initial state of order played no role in the mechanisms and kinetics of

recovery and recrystallization. It was inferred that the heavy cold deformation given to the samples destroyed the D0₃ order and significantly disturbed the B2 order. Grain boundary mobility was also not increased in the B2 structure compared to the D0₃ structure. It was concluded that there is a similarity in the mechanisms over the range of annealing temperatures examined and also irrespective of whether the material is B2 or D0₃ ordered.

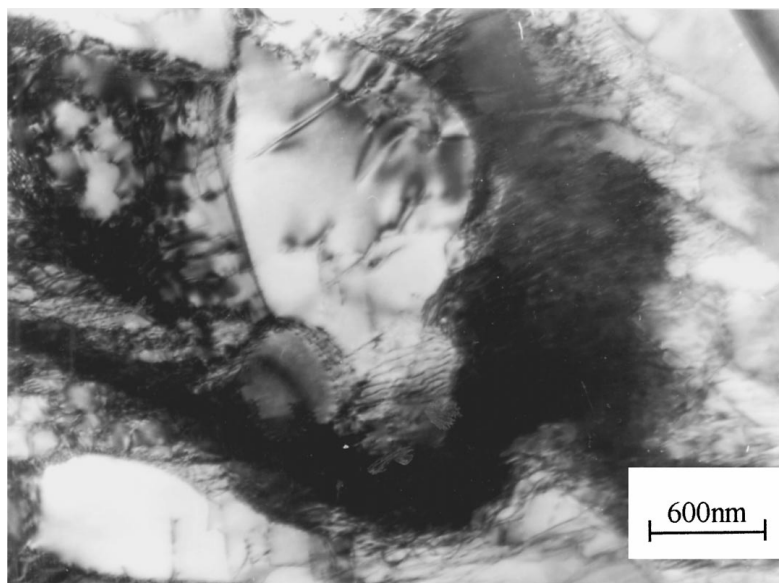
The present results showed that some of these conclusions could be extended to the three alloy compositions investigated and also to the behavior of these alloys near the b.c.c./B2 order-disorder transition. The weak composition dependence of RRG processes can be understood as follows [9, 15] as the Al content increases (a) the APB energy between the $\langle 111 \rangle$ partial dislocations



(a)

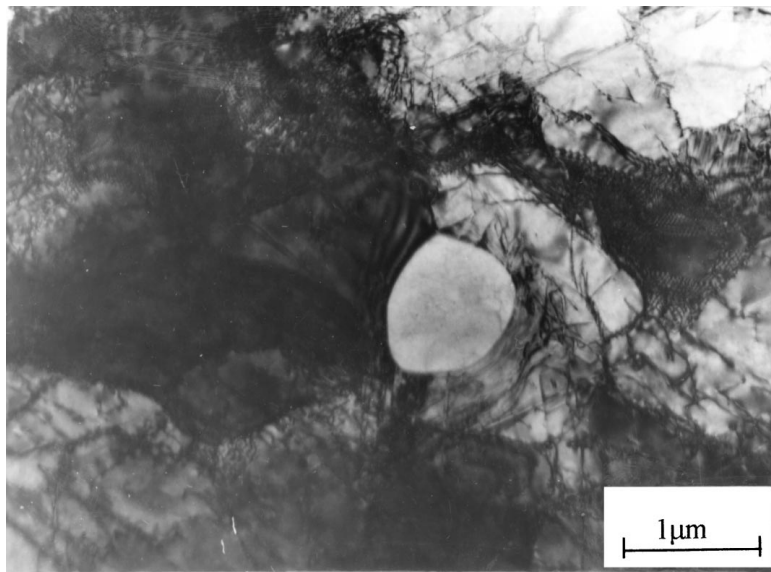


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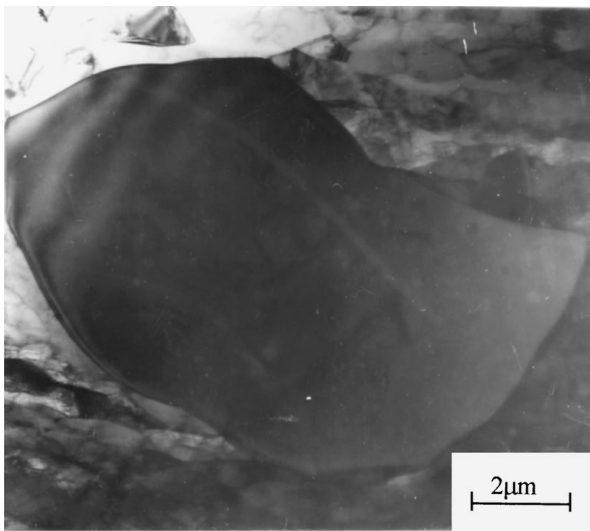


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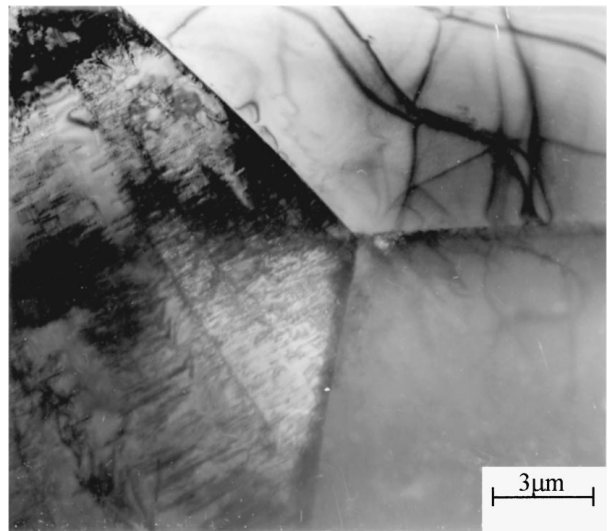
Figure 7 BF TEM micrograph of Fe-28Al annealed at 873 K (a) a linear dislocation array and subgrain formation can be observed, (b) square and hexagonal dislocation network formation, recrystallized grains with a high density of dislocations in the surrounding grain (d) equiaxed recrystallized grain found in a region with a high density of dislocations, (e) large strain free recrystallized grain adjacent to smaller abutting subgrains formed by polygonization, (f) BF TEM micrograph of Fe-28Al annealed at 1073 K, showing a triple junction formed after extensive grain growth. (Continued)



(d)

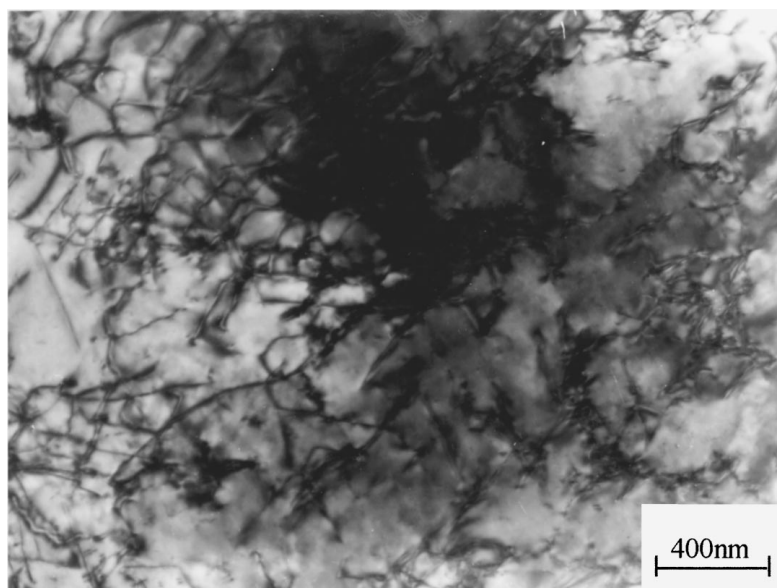


(e)



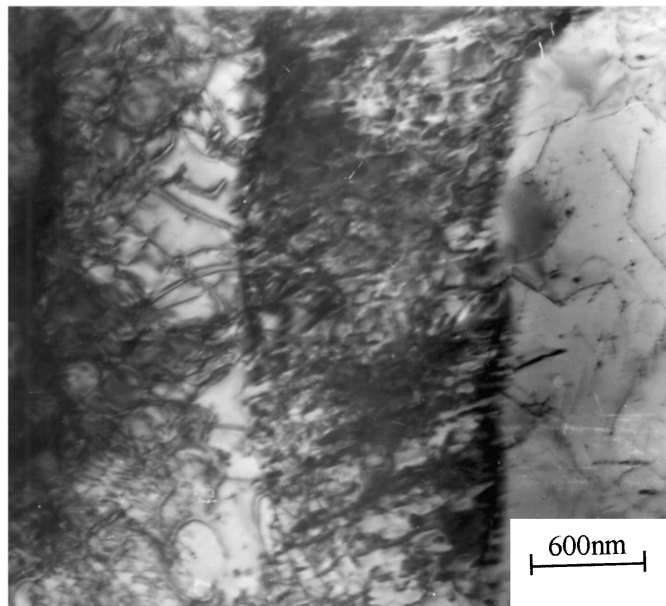
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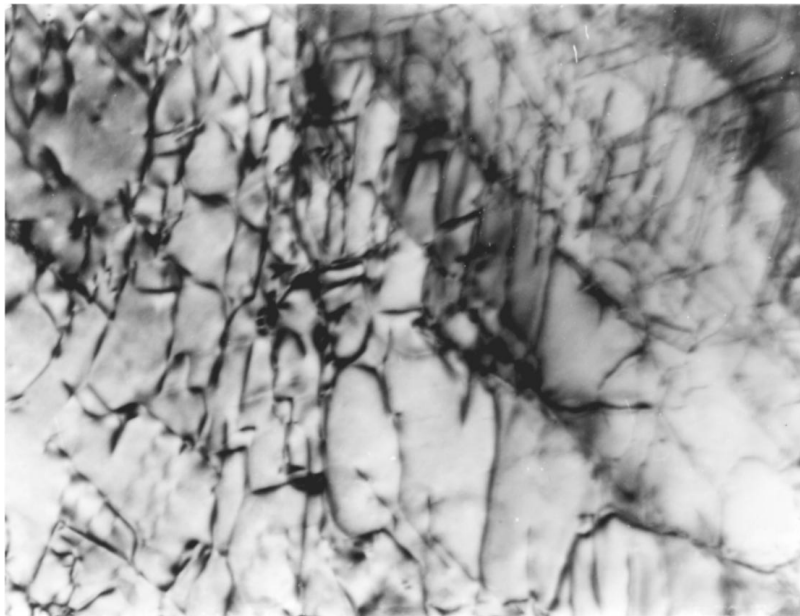


(a)

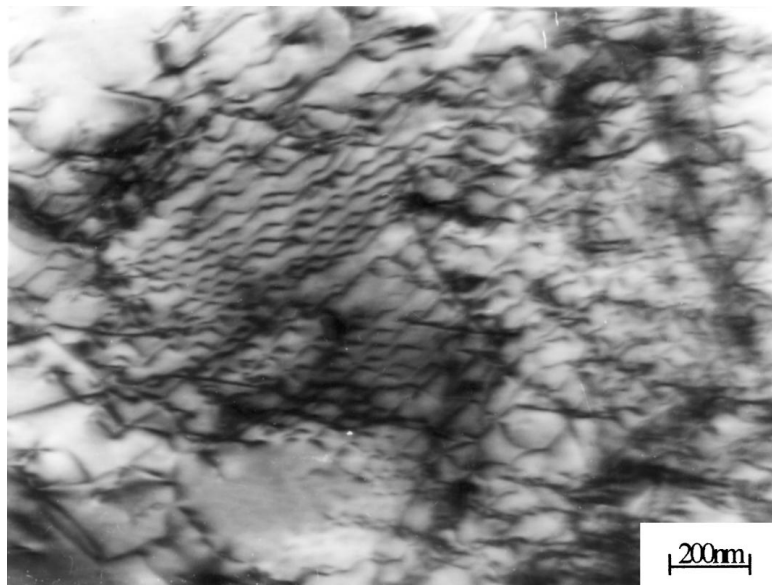
Figure 8 BF TEM micrograph of Fe-34Al in as-rolled condition, (a) showing single dislocations and dislocation tangles, (b) slip band formation, (c) loose dislocation network formation, (d) BF TEM micrograph of Fe-34Al annealed at 873 K, showing single dislocations and the early stages of dislocation network formation, (e) BF TEM micrograph of Fe-34Al annealed at 873 K, revealing well formed hexagonal dislocation nets, (f) BF TEM micrograph of Fe-34Al annealed at 973 K, extensive grain growth has occurred leading to the formation of high angle grain boundaries. (Continued)



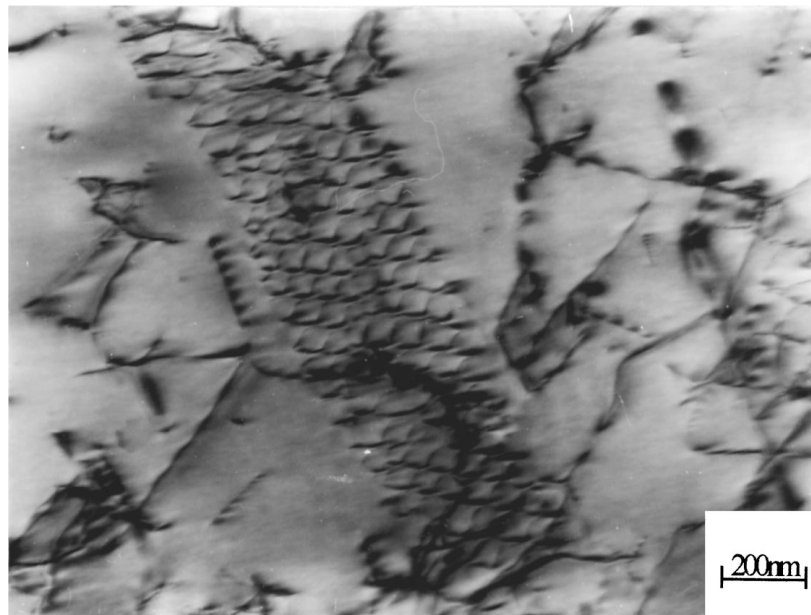
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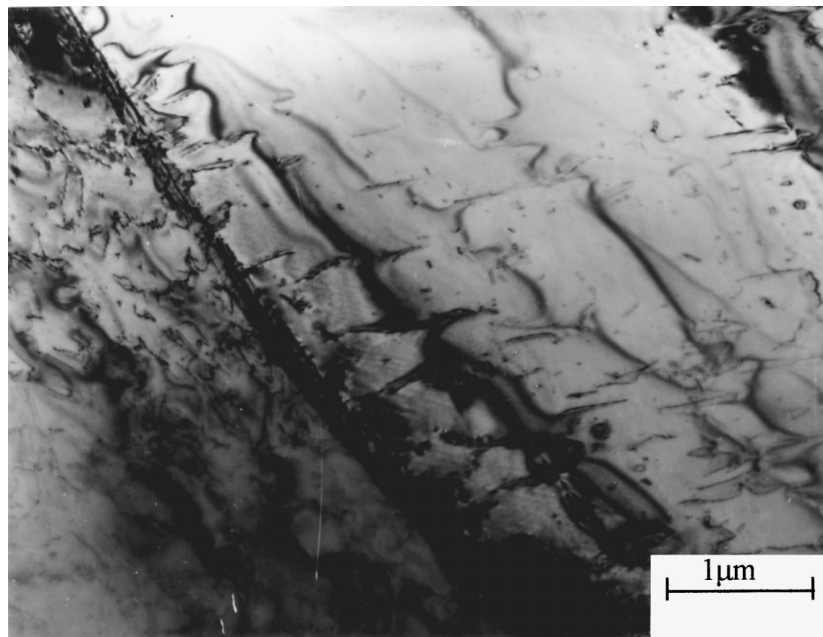
(c)



(d)



(e)



(f)

Figure 8 (Continued).

increases leading to easier dislocation climb and (b) the diffusivity decreases. Because of these opposing effects, the effect of composition is small.

The effect of annealing at 873 K is firstly, to reduce the average density of dislocations followed by the migration of single dislocations to form linear arrays. Dislocations in such arrays often show superjog formation. The nature of interaction of neighboring dislocations in such arrays depends on their Burgers vector and if the two dislocations have the same Burgers vector, mutual annihilation can take place. Alternatively, dislocations of the $\langle 111 \rangle$ type can interact to form dislocation segments. Such segments do not lie in the original slip plane and their Burgers vector is of the $\langle 100 \rangle$ type [17]. Both hexagonal networks such as those observed in iron and tungsten and square networks such as those seen

in Ta, can form by this process [18]. Both square and hexagonal networks have also been seen in semiconductor ErAs/GaAs heterojunctions grown by molecular beam epitaxy. Such III-V semiconductors with the NaCl structure are being extensively studied for novel device structures [19]. As in the case of tantalum, the size of such networks was distributed into two groups, some networks had a spacing of 200 nm and others with a spacing of 50 nm. These networks may have formed enclosed dislocation free regions which can act as recrystallized grains. Alternatively, low angle boundaries can form, resulting in subgrain formation. Subgrain migration can then take place by the migration of “Y-junction” nodes such as those observed in Fig. 4c and d. Annealing of the Fe-24Al alloy at 873 K also resulted in displacement fringes which may have arisen from

the formation of stacking faults, as in the case of Nb or W, or by the condensation of impurity atoms on {100} planes [20–22]. Further investigations are underway to resolve this issue.

Recrystallization was also observed at the TEM level in Fe-24Al and Fe-28Al alloys annealed at 873 K though the recrystallized grains were too small to be observed at the optical microscopy level. Recrystallized grains were formed with a large dislocation density difference between the two sides of the grain boundary of such grains. The presence of polygonized dislocation structures in the vicinity of such grains (Fig. 5a) suggested that this dislocation density difference was the driving force for recrystallization. This agreed with earlier results [18] that recrystallization occurred by the migration of boundaries which are formed by polygonization in regions with a large misorientation. Growth of grains by the migration of their boundaries parallel to themselves could also be inferred. In Fe-28Al, the recrystallized grains often showed a large orientation difference between the grains and the surrounding matrix [18]. The presence of large recrystallized grains surrounded by smaller subgrains suggested that polygonization produced high angle boundaries which are capable of migrating and forming large grains. Annealing at 973 K and higher temperatures did not yield much additional information on the mechanisms of recovery and recrystallization.

5. Conclusions

A study of Fe-24Al, Fe-28Al and Fe-34Al alloys was conducted to examine the recovery, recrystallization and grain growth processes in these alloys. Samples from these alloys, which were rolled and subsequently annealed at various temperatures, showed that:

1. From the TEM results, the mechanisms of recovery processes at 873 K could be inferred as: The glide and climb of single dislocations towards each other was followed by either annihilation or the formation of linear arrays. Neighboring dislocations in such arrays could interact to form either square or hexagonal dislocation networks. Subgrains were created by these dislocation networks acting as low angle boundaries. The migration of these subgrains can take place for example by the migration of appropriate Y-Junction nodes frequently observed in these specimens.

2. Recrystallized grains form either (a) by the enclosure of dislocation free regions by dislocation networks or (b) by the preferential growth of a few subgrains formed in the recovery process.

3. The effect of bulk alloy composition on the mechanisms of recovery, recrystallization and grain growth appeared to be weak in the temperature range investi-

gated. The effect of the B2-b.c.c. order/disorder transition temperature was also small.

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