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Effect of cold rolling on the mechanical properties of an FeCo–2V alloy

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The embrittlement of an FeCo–2V alloy, which is caused by ordering, has been found to be prevented or reduced by means of cold rolling over about 72% reduction. The specimens annealed after such rolling, in which lattice imperfections are virtually annihilated, do not always show embrittlement even after ordering. A simultaneous increase in strength and elongation, rather than an embrittlement, is brought about by the ordering at an early stage. This rolling effect has been shown not to depend on the following factors: existence of lattice imperfections, formation of textures, and changes in shape of grains. An attempt is made to explain the rolling effect in terms of the LCD zone model, local concentration-disordered zones, combining with an assumption that clusters composed of the composition near Co_3V are already formed in the course of the solidification: the clusters are, by the rolling, elongated and aligned along the rolling direction to form a fibrous structure, so that the same function as that in the fibre-reinforced materials would be induced by the forming of a ductile fibrous structure, which is produced around the individual elongated clusters as the LCD zones are developed by the ordering.

1. Introduction

The FeCo–X alloys are well known to embrittle proportionally with the advance of ordering, but recently it has been revealed that they do not always show embrittlement and yet exhibit a simultaneous increase in strength and elongation, if only severely cold rolled: shown by Pinnel and Bennett [1] in the FeCo–V alloy, and by the author in the FeCo–C alloy [2]. For intermetallic compounds, Shashkov *et al.* [3] have also pointed out a similar effect, which has been explained as follows: the segregation of interstitial impurities, including gaseous ones, on the boundaries and in the boundary regions of the grains must be continually reduced as the degree of plastic deformation grows. This effect may be termed the effect of deformation-induced dilution of impurities. Although the embrittlement of certain compounds may merely be explained by this effect, it has been suggested that in the FeCo–X alloys at least, the embrittlement may be interpreted only by a LCD model [2, 4, 5]: an improvement in the ductility can be brought about by the formation of the LCD zones which are built up in the vicinity of the individual particles of the precipitates of Co_3V compound.

The present work follows a previous report which has been carried out concerning the relationship between structural changes and mechanical properties [5], being performed to clarify the effect of cold rolling on the mechanical properties in the FeCo–2V alloys, in particular in respect to the interrelations among degree of rolling, structural changes, and changes in load–elongation curves. In addition, further development of the model will be attempted.

2. Experimental procedures

The specimens used are the same as those used in a previous report [5] and the composition consists approximately of 49.6%Fe–48.5%Co–1.75%V (wt%). Plates having the dimension of 5 mm in thickness, 30 to 60 mm in width, and 100 to 200 mm in length, were utilized for cold rolling to a 90% reduction. Prior to the rolling, the plates were heated at a given temperature for a given

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time, and then quenched in iced-brine water. In the case of cold rolling less than 90% reduction, the initial thickness was conditioned, by re-hot rolling or by machining, so that the finishing thickness of the plates to be cold rolled was constant, about 0.5 mm. Further detail on the procedures have been described in the previous reports [2, 4, 5].

Pole figures were measured by the Schultz back-reflection method with Co$K\alpha$ radiation. Standard samples for the measurement were made as follows: the filed powder of below 100 mesh was pressed, annealed at 800° C for 2 h, and then cooled slowly. The intensity for various specimens, for which Mo$K\alpha$ radiation was used, was measured under a condition with γ vibration.

3. Experimental results

3.1. Macroscopic structures and cracking in cold rolling

Three kinds of hot-rolled plates were cold rolled which were characterized by different structures,

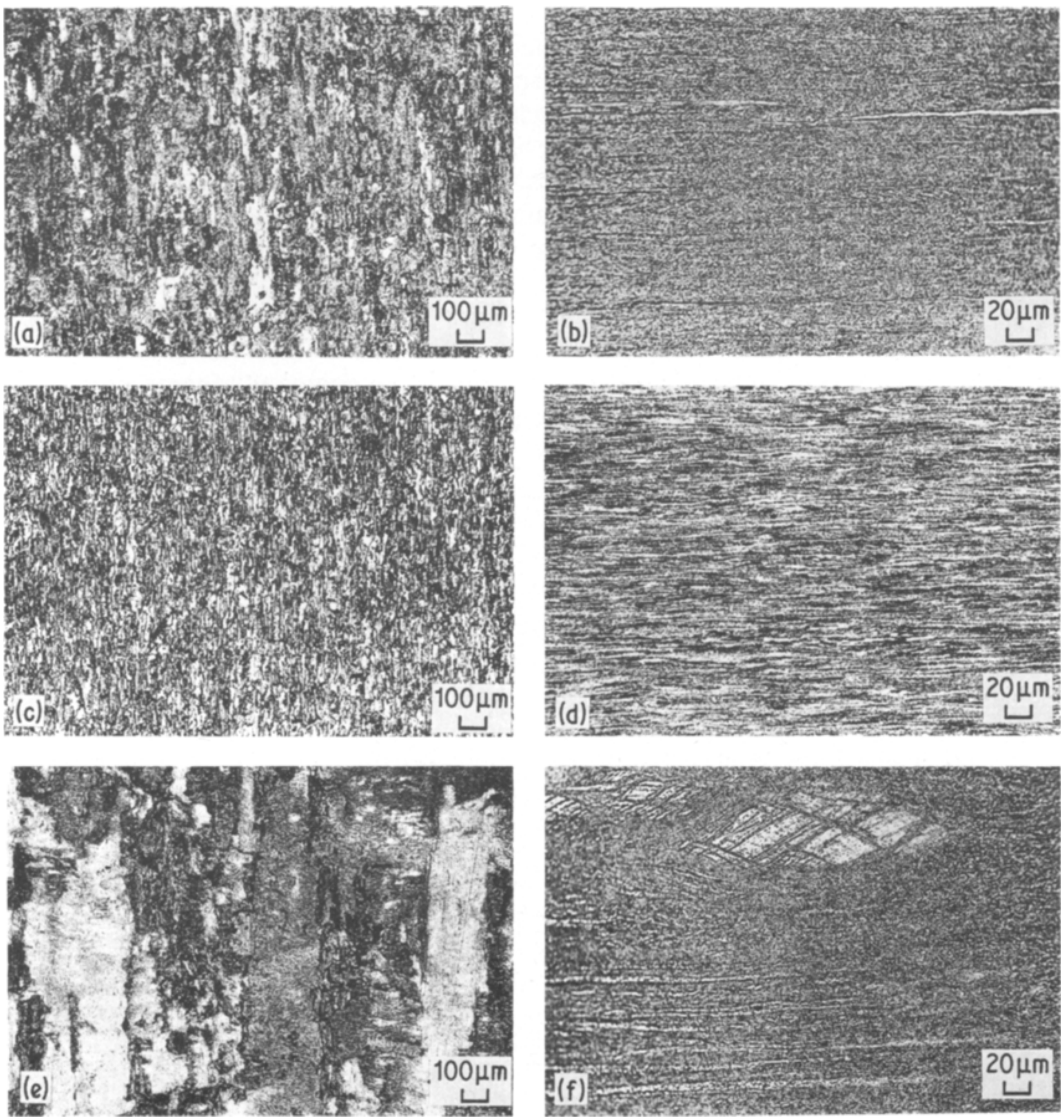

Figure 1 Micrographs in normal planes (a, c and e) and longitudinal sections (b, d and f) for specimens cold rolled to 90% after different conditions of annealing (aged at 650° C for 16 h to emphasize the features). (a) and (b) Martensite rolled after annealing from 1100° C, (c) and (d) mixed structure rolled after quenching from 950° C, and (e) and (f) ferrite rolled after quenching from 900° C.

i.e. (a) the martensitic structure obtained by quenching from the temperature where austenite phase exists singly, e.g. at 1100° C, (b) the mixed structure composed of martensite and ferrite, which is obtained by quenching from a temperature where the two phases, austenite and ferrite, coexist, e.g. at 950° C, (c) the ferritic structure obtained by quenching from a temperature where the stable phase is only ferritic, e.g. at 800 or 900° C; these structures have been illustrated in a previous report [5].

The ease of cold rolling of the plates depended on the previous structures preceding the rolling. The mixed structures could easily be cold rolled up to 90% reduction, with no cracking being observed. The two other structures, however, showed some cracking at an early stage of the rolling, e.g. at one or two phases, so that further rolling was prevented. But the majority of the fragments resulting from such cracking could be cold rolled to 90%, without further cracking. Cracking occurred more often in the ferrite plates than in the martensite ones.

Fig. 1 shows the structures of the three kinds of specimens which were cold rolled to 90% (all specimens were aged at 650° C for 16 h to emphasize this feature). Judged from the structures of the normal planes, characteristic differences can be seen in their uniformities; the most homogeneous structure is given by the mixed one, while the most heterogeneous, is given by the ferritic one. In the longitudinal section, such differences among the structures are small compared with that on normal planes. Differences in the occurrence of the cracking in cold rolling can be considered to correspond just to those in the uniform structures of normal planes; the specimen which consists of the most homogeneous structure is the most ductile. It can be said conclusively for the FeCo–2V alloy that at least at an early stage, heterogeneity of the structure plays an important role in the nucleation and the propagation of the cracks.

3.2. Effect of cold rolling on mechanical properties

Prior to tensile tests, the relationship between ordering and ageing was examined. In Fig. 2 the advancement of the ordering occurring in different temperatures are shown with respect to the cold rolled specimens which consist of three kinds of different structures as mentioned above. In ageing at 500° C, the degree of ordering can be seen to rise gradually, independent of differences in the quenching temperatures, namely, of those in the structures of the specimens. In ageing above 600° C, the rates of the ordering are higher than

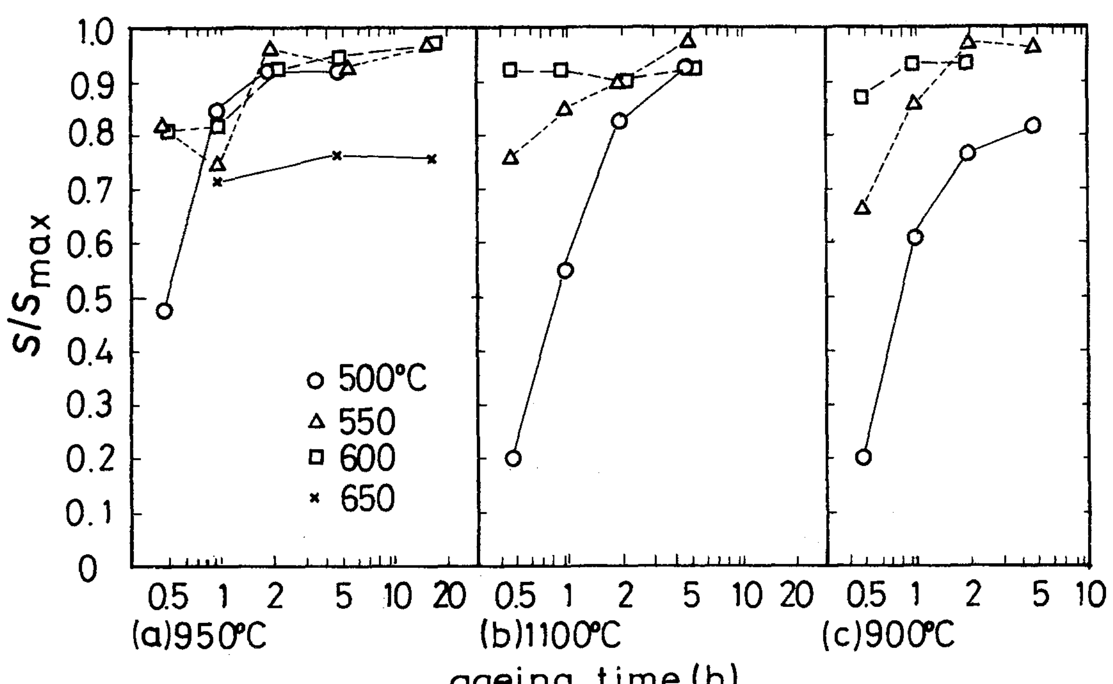

Figure 2 Effect of ageings on degree of ordering.

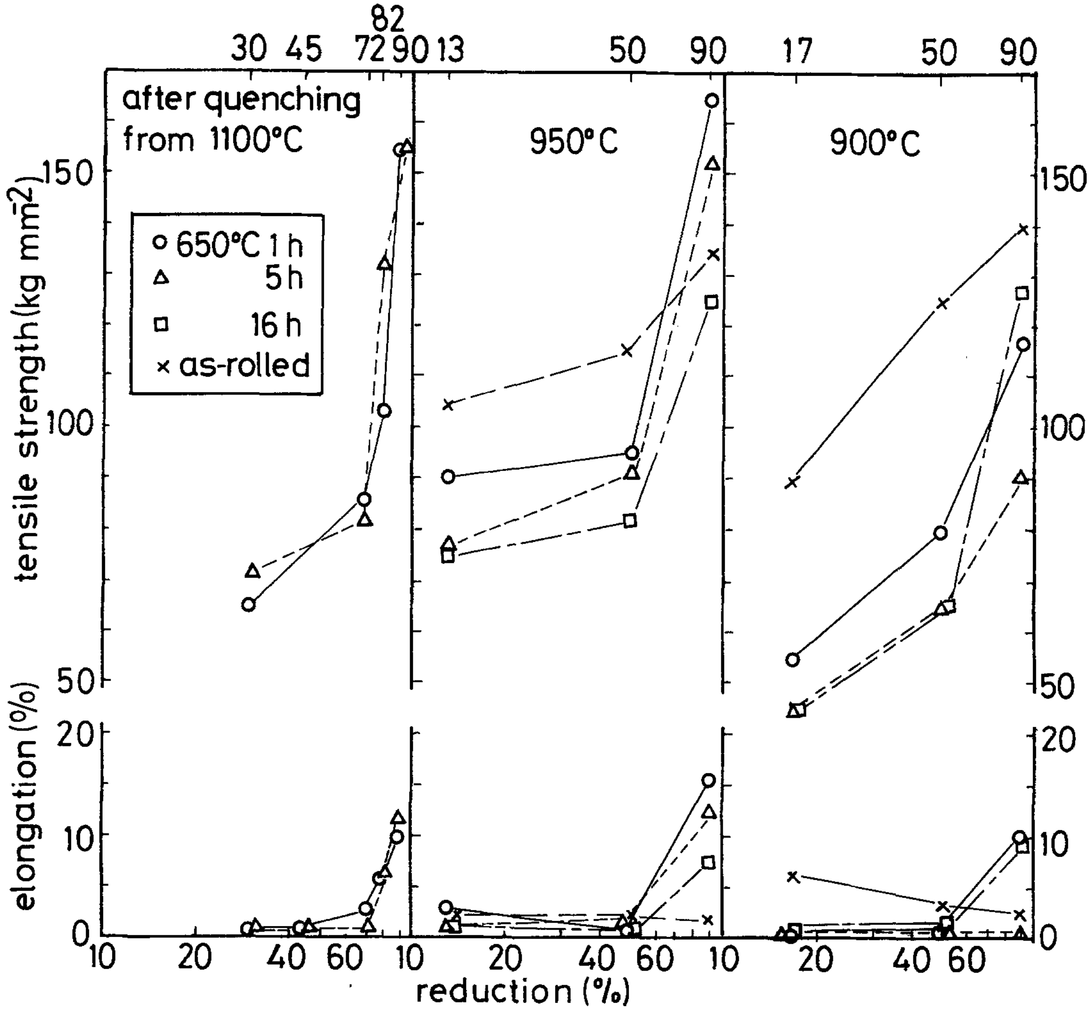

Figure 3 Effect of rolling reduction on improvement of mechanical properties.

those at 500° C, and about 80% of a saturated value is reached for a short time, i.e. no more than 30 min. From the results, an ageing rate at 400° C, which will be used in a subsequent section, is easily predicted to be much lower than that obtained at 500° C. In addition, the results of these ageings are in good agreement with other data [6–20].

In Fig. 3 effects of cold rolling on the mechanical properties are shown with regard to the three kinds of samples. In the case for a low degree of rolling, all the specimens are brittle if only ordered, but elongation begins to appear when a critical reduction is reached, for example, from about 72% for the case of the martensitic structures. It can be seen that, in the high reduction over the criticalness, the ordering does not always bring about embrittlement and yet shows a simultaneous increase in strength and elongation. The critical values in other structures may also be presumed to be located about from 70 to 80% reduction. In a previous report [5], all the specimens for which no cold rolling are carried out were always brittle even in the specimens that were heat-treated so as to obtain more fine and homogeneous grains, whenever they were aged. It can be concluded, however, that the embrittlement is reduced or excluded only by a severe cold rolling, irrespective of differences in the structures and of those in the degree of the ageing.

Within the limit of thin foil observations, each of the specimens rolled about 50% has similarly shown a complex structure with numerous dislocations. As an example, the martensite rolled to 90% is shown in Fig. 4. Such a structure readily disappeared on heating at 800° C for 1 min. Fig. 5 indicates an example of heating for 2 min, with no dislocation being observed; further annealing at

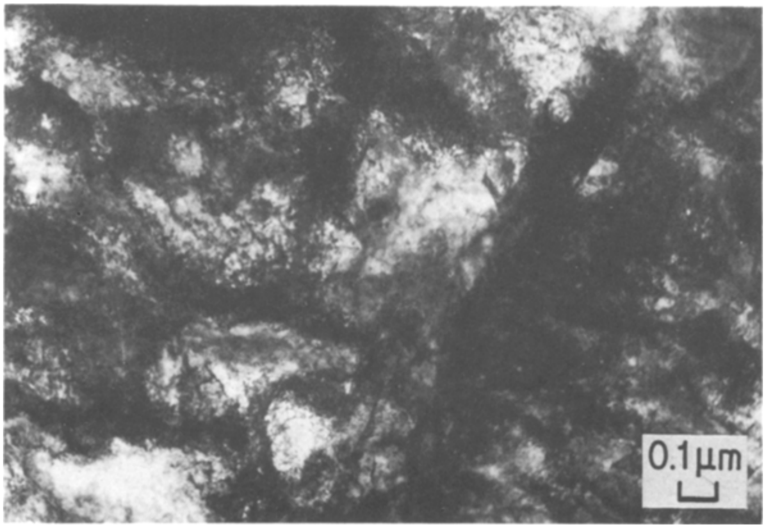

Figure 4 A thin foil micrograph of martensite cold rolled to 90%, showing a complex structure consisting of numerous dislocations.

800° C during 1 h was merely accompanied by grain growth.

3.3. Load–elongation curves

The changes in load–elongation curves of the specimens tested show some interesting phenomena, in addition to changes in strength and elongation. As are shown in Fig. 6, the curves are roughly divided into six classes for convenience.

Curve 1 was always shown by very brittle specimens, i.e. (a) as-cast, (b) as-hot-rolled, (c) both states aged after the cold rolling of less than the critical reduction. Curve 2 was given by specimens quenched from the high temperature, i.e. from 800, 950, and 1100° C, which correspond to the disordered states without cold rolling. The specimens were ductile and the elongation were large. Curve 3 was observed for cold-rolled plates. Such specimens were composed of high strength and large reduction in area, but their elongations were small because of the necking due to plastic instability. Curve 4 developed when the specimens cold rolled over the critical reduction were aged; the work hardening appeared after yielding, without the occurrence of Luder's banding. The elongation was large but no necking was observed on breaking. Curves 5 and 6 could be seen in the specimens which were cold rolled over the critical reduction and then aged or re-annealed. Luder's bands appearing in the specimens happened instantly after yielding. In the case for sufficient annealing after cold rolling, e.g. at 800° C for 2 min, Curve 5 was observed; in the case for the specimens ordered after such treatment, Curve 6 was obtained. Curve 5 had a tendency to approach Curve 6, with an increasing degree of ordering.

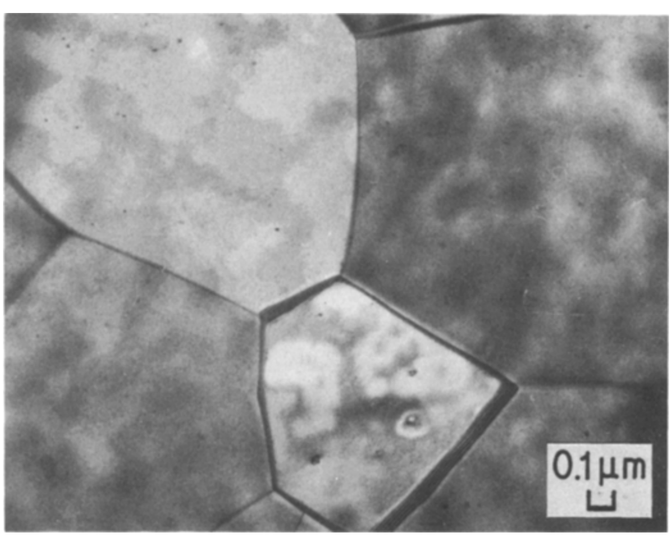

Figure 5 A thin foil micrograph of martensite annealed at 800° C for 2 min after cold rolling to 90%, showing a dislocation-free structure.

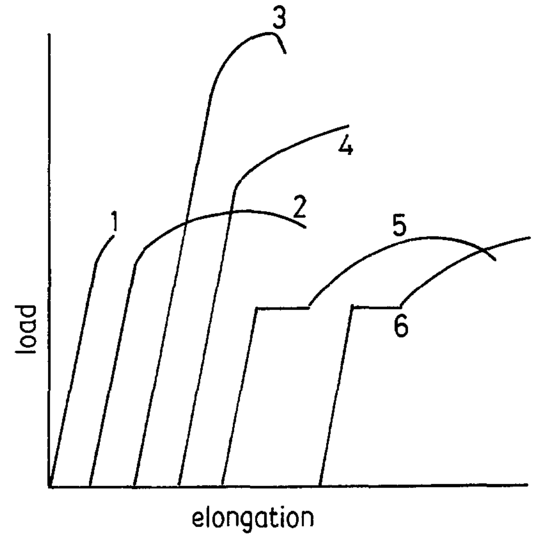

Figure 6 Load–elongation curves occurring on tensile tests for specimens subjected to various heat treatments and cold rolling.

Curve 6 was the same as Curve 5, except that no necking occurred in the former.

The observations on load–elongation curves have been made [1, 21–25]. Pinnel and Bennett [1] have shown that the curve similar to Curve 6 was produced when the cold-rolled specimens were aged. However, they did not find that the important condition for the occurrence of Luder's bands is deformation over a critical reduction. Luder's bands could not be observed in specimens that were not cold rolled, but only in those that were cold rolled and then aged or annealed. As shown in Fig. 5, no imperfections due to the rolling are observed in treating under such condition, as in the quenched specimen, without cold rolling, from above 800° C. The fact that the banding only occurred for the rolled specimens, suggests that any rolling effect remains. The occurrence of the bands, therefore, can be used for checking whether or not any effect of cold rolling remains after an annealing. Interestingly, even after annealing at 800° C for 1 h the bands can always be seen, and the samples annealed at 1050° C for 5 min, which produced Curve 2, again showed Luder's bands if they were again annealed at 800° C. It follows from the evidence that the effect of cold rolling may not disappear only by applying the heat treatments at 1050° C for 5 min or at 800° C for 1 h, although the structures have been changed drastically by these treatments, as can be understood by comparing Fig. 5 with Fig. 4.

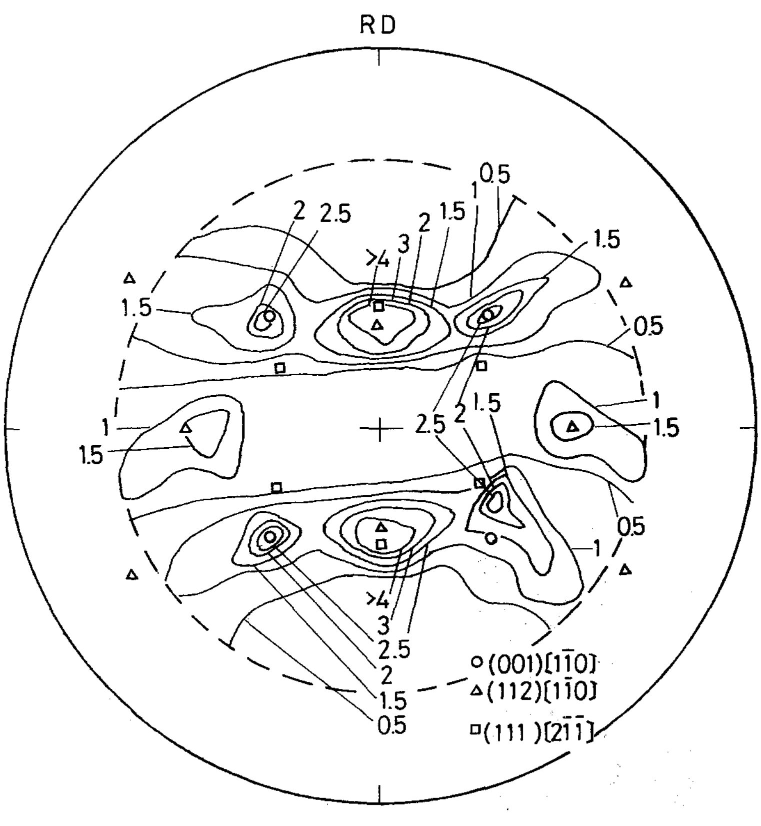

Figure 7 A (110) pole figure of martensite cold rolled to 90% after quenching from 1100° C.

3.4. Textures

Investigations concerning the texture of FeCo–V alloys have been reported [26–28]. It is generally said that changes in texture of these alloys are almost identical with that of the iron-based bcc alloys: in the case of 90% cold rolling, the main component is found to be $(001)[1\bar{1}0]$, $(112)[1\bar{1}0]$ and $(111)[2\bar{1}\bar{1}]$; with the recrystallization of the rolled specimens, the main component is occupied by $(111)[2\bar{1}\bar{1}]$ and the minor by $(000)[1\bar{1}0]$ or $(112)[1\bar{1}0]$ [26–28].

Fig. 7 shows a (110) pole figure of the massive martensite cold rolled to 90%; three strong components, namely, $(001)[1\bar{1}0]$, $(112)[1\bar{1}0]$, $(111)[2\bar{1}\bar{1}]$ are observed; these components are also well known in the iron-based bcc alloys. Fig. 8 is a (110) pole figure of the specimen subjected to a martensitic transformation by quenching from 1050° C after cold rolling to 90%. A component, $(001)[1\bar{1}0]$, can be seen to decrease by this transformation, and this tendency can be very clearly understood from the result of the measurement for the integrated intensity. In Fig. 9, the result is shown for the plates subjected to different processes. Plates which were cold rolled to 90% after quenching from different temperatures are, including a plate aged at 700° C for 16 h, quite similar to one another in intensity values, the components being essentially only three: (100), (111), (211). A plate, however, which was subjected to a transformation after cold rolling, shows a tendency to allow the texture produced by the rolling to randomize, as is indicated on the right hand.

Fig. 10 shows the integrated intensity of the plates used in the previous paper for FeCo–C alloys [2]. The plates were decarburized at 1050° C for 10 h after cold rolling to 90%, so that the grains had grown sufficiently. The plate with 2% carbon is almost composed of (100) planes, while the plate with 0.5% carbon consists of (100), (211), (311) and so on, though the intensities are weak. This difference in the component between both plates is not considered to be due to the differences in carbon contents, and presumably it may be merely a result of an accidental coarsening having occurred in the individual grains. Although those pole figures were measured using discs of 34 mm diameter with 0.5 mm thickness, a small number of grains alone were included in the tensile test pieces consisting of a width of 2 to 3 mm, so that it is questionable whether the effect of texture must be applied to such pieces. In addition, since the pieces that had large grains and caused the embrittlement after decarburization

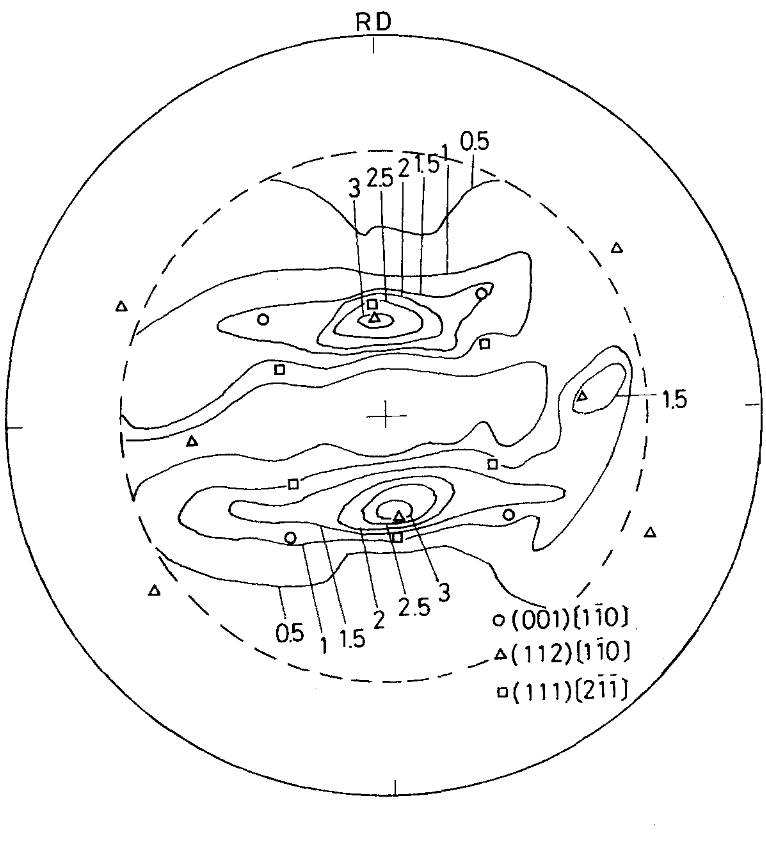

Figure 8 A (110) pole figure of martensite obtained by quenching from 1050° C after cold rolling.

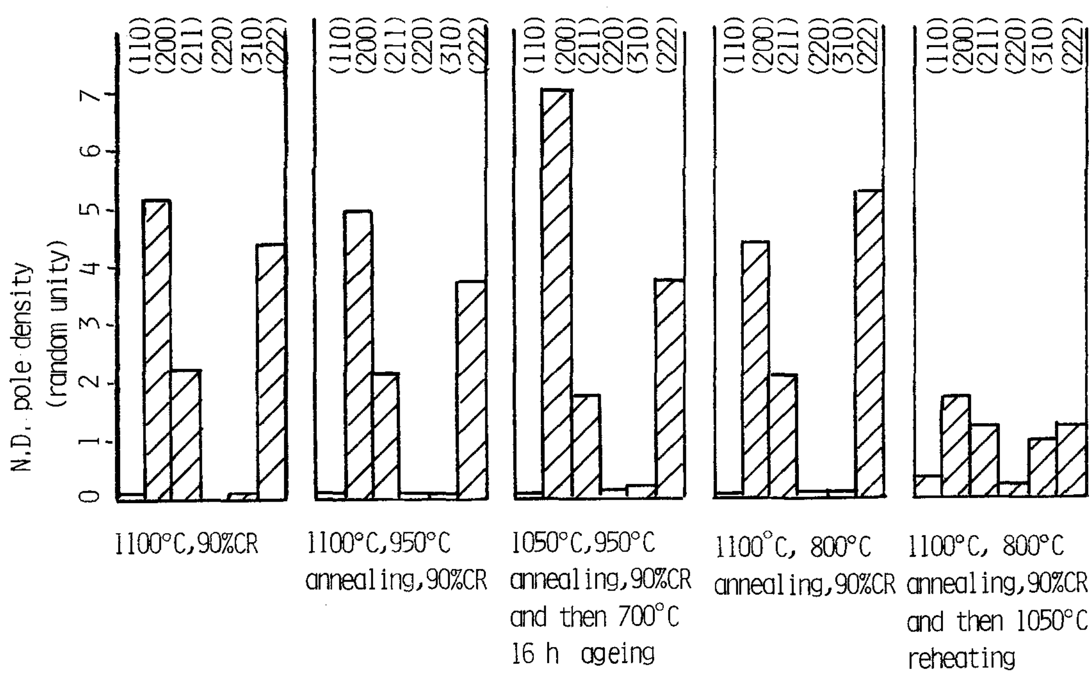

Figure 9 Integrated intensity for different situations of an FeCo–2V alloy.

caused recovery in ductility, only if they had again been carburized as has been shown previously [2], it is concluded that no texture directly governed the brittleness and ductility in FeCo–C alloys.

3.5. Increase of strength and elongation occurring after ordering

As stated in the preceding section, the effect of cold rolling has been observed to remain even after annealing at 800° C. Since the imperfections produced in the rolling can be essentially annihilated by annealing, the annealed specimen, if ordered, is therefore expected to cause an embrittlement.

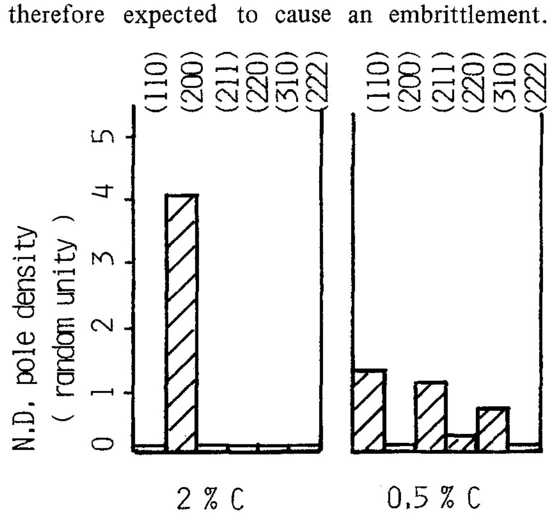

Figure 10 Integrated intensity for FeCo–C alloys decarburized at 1050° C for 10 h.

Interestingly, a ductility, as opposed to the expectation, occurs at an early stage in the ordering treatment. In Table I the results are shown, in which three kinds of rolled specimens are indicated with regard to tensile properties caused by ageing at 400 and 600° C after annealing at 800° C for 2 or 5 min. The orderings after the annealings are accompanied by an increase in strength and in elongation. The mechanical properties of the specimens annealed at 800° C are inferior to that subsequently aged at 400 or 600° C, a peak being at 1 h in ageing at 400 and at 30 min at 600° C. Ordering leads to an excessive increase, not a decrease, in tensile properties. In the early ageings no change in structure is detected, though further ageing may bring about plate-like precipitates, as shown in a previous paper [5].

Table II shows the effect of annealing time and of transformation upon the properties. In all cases which are annealed for 1 h at 800° C, transformed after quenching from 1050° C, and annealed after the transformation, favourable results are induced, similarly, at early stages in the ageings. From the results, it can be denied that a specified configuration of dislocations may be responsible for improving the ductility of FeCo–V alloys [29, 30]; such dislocations, necessary to form such a configuration, are not included already in the annealed specimens.

TABLE I Effect of pre-annealing at 800° C for short times after cold rolling upon mechanical properties of ordered specimens

Initial conditions	Annealing conditions	Ageing (° C)	Mechanical properties	
			Elongation (%)	Tensile strength ($kg\,mm^{-2}$)
1100° C, 98%CR	800° C for 5 min	as annealed	9.0	79
		400 1 h	11.5	101
		400 16 h	5.0	70
		400 48 h	7.0	75
	800° C for 2 min	as annealed	10.0	77
		600 0.5 h	11.3	104
		600 1 h	6.9	87
		600 5 h	6.3	83
950° C, 90%CR	800° C for 5 min	as annealed	8.5	82
		400 1 h	10.0	86
		400 16 h	5.0	75
		400 48 h	5.0	68
	800° C for 2 min	as annealed	12.5	103
		600 0.5 h	18.8	140
		600 1 h	12.5	123
		600 5 h	6.3	73
800° C, 90%CR	800° C for 5 min	as annealed	10.0	90
		400 1 h	11.0	96
		400 16 h	6.0	73
		400 48 h	5.0	67

4. Discussions

It has been revealed in the previous experiment [5] that, even if the structures of the FeCo–2V alloy were conditioned so as to be as fine and uniform as possible, ordering treatments caused such specimens to embrittle. It has been found in the present experiment that, if only cold rolled over about 72% reduction, the specimens show ductility even after ordering. Hereafter this effect will be called the rolling effect.

Except for Shashkov's dilution effect, the increase in deformation is accompanied by the following variation. Firstly, lattice imperfections, such as dislocations, vacancies, and so on, are introduced, occasionally with deformation bands or twinning, so that complex structures may be

TABLE II Effect of prior annealing conditions upon mechanical properties of ordered specimens

Initial conditions	Annealing conditions	Ageing (° C)	Mechanical properties	
			Elongation (%)	Tensile strength ($kg\,mm^{-2}$)
1100° C, 90%CR	800° C for 1 h	as annealed	10.0	81
		400 1 h	11.5	90
		400 16 h	7.0	78
		500 1 h	6.0	70
	1050° C for 5 min	as annealed	12.0	75
		400 1 h	13.5	81
		400 16 h	9.5	96
		500 1 h	4.0	74
	1050° C for 5 min and then 800° C for 5 min	as annealed	10.5	79
		400 1 h	16.0	90
		400 1 h	4.0	61

produced. Secondly, textures can be formed. Thirdly, changes in the shape of grains occur, in general the grains being elongated along the rolling direction. Fourthly, if any phases, e.g. secondary ones, exist previously in samples, changes in the distribution of the phases are caused; the mean path among the phases is enlarged in the rolling direction but shortened in the normal direction; the distribution, after a while, becomes more homogeneous and more dense with increasing rolling reduction.

The rolling effect may, in the first place, be suspected to be associated with the lattice imperfections, but the problem can be removed by the fact that the effect has occurred also in specimens annealed at 800° C for 2 min as well as 1 h after cold rolling, because in such specimens the imperfections could almost be annihilated by the heat treatment. The question on textures can also be eliminated in view of the following phenomena: the rolling effect, whenever the specimens have been subjected to cold rolling over the critical reduction, has always occurred similarly even in different textures which could result from different treatments, e.g. decarburization, transformation, and annealing. The shape change in grains also is not considered to relate to the rolling effect, because the effect took place in a specimen which was subjected to a transformation after the cold rolling, in which such elongated grains were changed to form equiaxed grains. The rolling effect can accordingly be understood to be independent of the three factors so far mentioned, i.e. imperfections, textures, and shape of grains.

The fact that an increase in ductility, even after ordering, develops in the specimens which were cold rolled over the critical reduction, is forced to assume that a certain kind of cluster already exists in the specimens, in addition to the application of the LCD model. Clusters have been known also in the FeCo–V alloys [19, 32–34], but the clusters have been shown to form in the solid solutions. To account for the rolling effect, however, it must be assumed that a certain kind of cluster could already occur in the course of solidification of the alloys. The assumption is based on the phenomena: (a) for ductility after ordering there is a difference between the specimen that was quenched from 800° C as it was cast and that which was cold rolled over the critical reduction and then was again quenched from 800° C; the former was brittle while the latter ductile, respectively, and (b) the effect of cold rolling is retained even after sufficiently annealing, as has been shown in the load–elongation curve. These facts could not be argued without the existence of clusters, though there is a question whether the clusters are formed either in the state of a solid solution or in the course of a solidification. If the clusters are formed in a solid solution, then in both specimens mentioned above the occurrence of the same ductility must be expected, because the same condition for the nucleation of the clusters is considered to be provided in both solid solutions. In fact the cold rolling is essential to improve the ductility of the alloy. From these facts it is suggested that the clusters are formed in the course of a solidification process. In any event, the fourth factor so far made should be considered anew, in which many of the phenomena could be interpreted easily.

The distribution of such clusters is though to be dilute and heterogeneous in the as-cast matrix, but it tends to become more dense and more homogeneous with increasing rolling reduction. Since the second phases existing in a specimen, if plastic, are increasingly elongated along the rolling direction with increasing cold rolling, the specimen thus obtained can be regarded as a fibrous material, which comprises of the situation that the brittle fibres may be reinforced by the ductile fibres resulting from the formation of the LCD zones. This fibre-induced effect is well known in the field of composite materials. If the volume fraction and the density of such second phases are constant, a reduction more than a critical value may be required for the purpose of developing such an effect. In the present experiment the critical value is about 72%, which is considered to be evidence of such a mechanism. This value may practically be varied, depending on the factors, such as, cooling conditions of the ingots, quantity of additional elements, and so on.

With an increasing degree of ordering, the precipitate, composed approximately of CO_3V, has been observed to occur [5, 19, 31], and its distribution occurring in ageing is found in the previous paper [5] to exhibit a remarkable difference between the as-cast state and as-cold-rolled state: the distribution is dilute and dense, respectively. Considering that the rolling effect is never obtained with the specimen not cold rolled, and that it is always shown by the cold-rolled specimen even after they are annealed sufficiently prior to ordering, it is likely that a certain kind of cluster, presumably consisting of a composition near a Co_3V compound, is produced in the course of the solidification, and that the clusters are relatively stable remaining unchanged even after annealing. On combining such assumptions with the LCD model, the rolling effect may be explained: that the effect is not observed in the as-cast state is due to the distribution of the LCD zones formed around the individual clusters being dilute, while it is observed in the state cold rolled over the critical reduction is because the fibre-induced effect caused by the fibrous structure resulting from the rolling is generated anew. Although in the specimens, which were cold rolled to 90% and then annealed at 800° C, load–elongation curves with Luder's banding is obtained in the tensile test after they are aged, the occurrence may be regarded as evidence for the assumption. The occurrence is surely attributed to the cold rolling, but variations of, for example, lattice imperfections, grain size, grain shape, and texture, have been shown not to relate, as mentioned above, to this occurrence. If there are stable clusters in the as-cast state, then shape change of the clusters will occur by such a rolling, except for the variations mentioned above; there is a possibility of elongated LCD zones aligning fibrously and of a fibre-induced effect developing, depending on the volume fraction of the clusters and on the rolling reduction.

Assuming the clusters, consisting of the composition near Co_3V, already to be formed in the course of the solidification, the effect of vanadium addition upon improving the mechanical properties of FeCo–V alloys can be considered to be as follows. By the addition of the element, vanadium, the clusters having the composition near Co_3V are already formed during solidification; the LCD zones are also produced always in the vicinity of the individual clusters; the degree of disordering resulting from quenching is facilitated by the formation of the zones, so that under the same cooling condition the ductility is obtained more easily in the sample with vanadium than in that without vanadium. An increase in the volume fraction of the clusters, e.g. by more addition of vanadium, and that in the density of the clusters formed during the solidification, e.g. by controlling the solidification conditions, are required in order to allow the ductility of the samples to be obtained more easily. In addition to the formation of the LCD zones resulting around the clusters, a fibre-induced effect is combined by a cold rolling, and in the present experiment a favourable condition is considered to be developed from about 72% reduction.

5. Conclusions

The specimens cold rolled over a critical reduction, about 72%, have not always exhibited embrittlement, even though ordered after annealing. This rolling effect is confirmed to be independent of certain factors, i.e. lattice imperfections, textures, changes in shape of grains. Occurrence of the rolling effect has been explained, as follows, on the assumption that the clusters consisting of the composition of about Co_3V are already formed during the solidification: (a) since there are the possibility of LCD zones being produced around the individual clusters, the volume fraction of disordered zones resulting from quenching is more in the samples with vanadium than in those without vanadium, thus the ductility can be obtained more easily; (b) since the clusters are, by cold rolling, elongated along the rolling direction, a fibre-induced effect, which is attributed to the elongated LCD zones emerging around the elongated clusters when the samples are subjected to ordering, is developed over a critical reduction of rolling, the embrittlement due to ordering can be decreased or prevented by the effect.

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