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Structures and mechanical properties of an FeCo–2V alloy

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Effect of structural changes based on heat treatments upon the ductility of an FeCo–2V alloy has been studied. The ductility does not always result from the martensitic transformation, but it always results from the ferritic structure which is composed of large and heterogeneous grains, whenever the specimens are quenched from a temperature of the ferritic region. In the case of the specimens heat treated at and quenched from a temperature where two phases can coexist, the ductility is strongly affected by the uniformity of the mixed structure obtained by such a treatment. The ductility of the mixed structure is improved by combinations of various heat treatments. After ordering, however, all specimens treated have never shown any ductility. The distribution of the precipitates occurring during ordering have shown a remarked difference between as-cast and as-cold-rolled specimen; the former is dilute, while the latter is dense. The composition of the individual particles extracted from the samples which are satisfactorily aged are shown to be about 64Co–21V–15Fe on average. Following to the information, a possible mechanism of improving the ductility of the alloys is argued on a model of local concentration-disordered zones.

1. Introduction

Equitatomic FeCo alloys or those near the composition are too brittle and weak to be fabricated, but it has been found that the workability of the alloys can be improved by addition of vanadium [1] and chromium [2]. The mechanism, however, has yet to be clarified, in spite of many investigations. An approach to revealing the effect of the addition is to understand the relationship between the structures and the mechanical properties of the alloys. Although some studies on the relationship have been reported hitherto [3–14], the observation of the structural changes have been made only for the states that are in the course of the process, i.e. for the states hot-worked. Therefore, there is a question that the variables necessary to ascertain the relationship between structural changes and mechanical properties are insufficient.

The present investigation has been carried out for the as-cast and as-cold-rolled states, in addition to as-hot-rolled states, showing some phenomena thought to be useful to understanding the mechanism of effectiveness brought about by additional elements. A report concerning the effect of cold rolling will be given elsewhere [15].

2. Experimental procedures

Materials consisting of electrolytic-iron and -cobalt and ferro–vanadium alloy, were melted in vacuo, and were cast into a 17 kg ingot with a section of 90 mm square. The chemical composition of the ingot is as follows: Fe 49.6 wt %, Co balance, V 1.75 wt %, C $<$ 0.005 wt %, Si 0.048 wt %, Mn 0.017 wt %, P 0.005 wt %, S 0.008 wt %, Ni 0.073 wt %, Cu 0.01 wt %, Sn $<$ 0.001 wt % or Fe 51 at %, Co 47 at %, V 2 at %.

The ingot, from which some specimens with 1 mm in thickness were cut directly to study the as-cast state, was heated at 1200° C for 4 h and hot rolled to plates with thickness of 1 to 5 mm and with width of 30 to 60 mm. Plates with 5 mm were submitted to the cold rolling to 90%, after iced-brine quenching from the different temperatures.

Tensile tests were carried out at a crosshead

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rate of 1 mm min^{-1}, using the specimens consisting of dimensions with a thickness of 0.5 to 1 mm and a width of 2 to 3 mm and with gauge length of 20 mm.

Heat treatments were achieved in a salt bath for the temperatures less than 700° C and in a tube furnace with streaming argon gas, for temperatures higher than 800° C.

A solution of 5% nital was used to observe structural changes and to extract, by a conventional method, the precipitates which occurred during ordering.

The observations by transmission electron microscopy, the specimens for which were prepared by a 95%CH_3CHOOH–5%$HClO_4$ solution, were performed with a H-500 microscope, operating at 125 kV. The identification of the extracted precipitates was made by H-700H analytical electro microscope attached with Kevex's energy-dispersive spectrometer, operating at 200 kV.

Transformation temperatures were measured, using a device for thermal expansion. They were determined both for a heating and cooling rate of 1200° C h^{-1}.

3. Experimental results

3.1. Heat treatments and structural changes

Transformation points were measured prior to various heat treatments. The results are as follows: the starting temperature of a ferrite to austenite transformation is about 925° C and the finishing temperature is about 980° C; conversely, the starting temperature of an austenite to ferrite transformation is about 910° C and the finishing temperature is about 840° C. In the temperature range from 925 to 980° C, two phases, namely, ferrite and austenite, can coexist, and the volume fraction of each phase is affected by heating temperatures and times in the range, and the previous structures of the specimens. In the temperatures above 980° C and below 840° C, austenitic or ferritic phases exists singly, respectively, though in the ferrite an ordering can occur below about 730° C.

The existence of a transformation can make it possible to bring about various structures, and many reports relating to the structures have been presented hitherto. A massive martensitic structure, which can be obtained by quenching from a temperature where the austenitic phase occurs, has been observed by an optical method [3–8] and on thin foil [7, 9]. The mixed structures obtained by quenching from the coexistence temperature of two phases are also confirmed by optical observations [7, 8, 10–14] and on thin foil [9]. However, the effect of processes preceding heat treatments on structural changes is not yet known.

For the purpose of understanding such an effect, three kinds of states were used, i.e. as-cast, as-hot-rolled, and a massive martensitic one. As shown in Fig. 1, there are differences in the structures between as-cast and as-hot-rolled specimens, the former being composed of large and heterogeneous columnar grains of the order of a millimetre while the latter fine and homogeneous grains are of the order of a micrometre. Fig. 2 exhibits a massive martensitic structure with numerous dislocations, which can be obtained by quenching from above 980° C in the case of the present alloy. Since the three states, i.e. as-cast, as-hot-rolled, and as-quenched, consist of characteristic structures, respectively, it is expected that the differences will affect their mechanical proper-

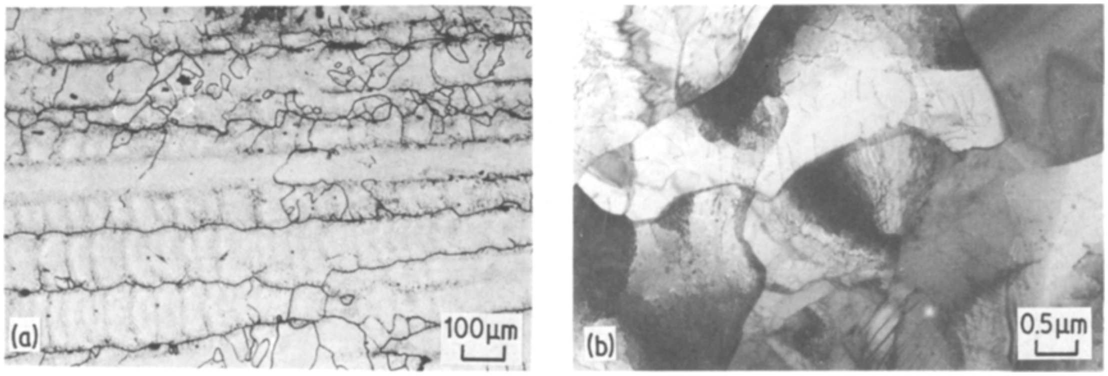


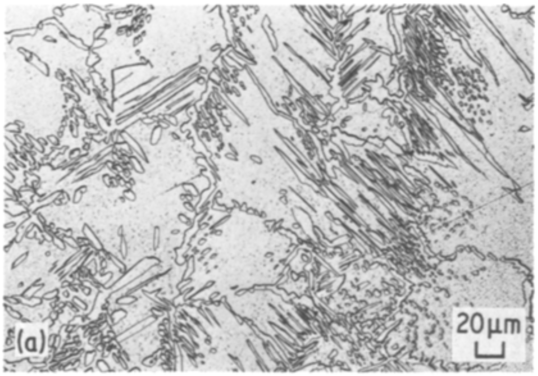
Figure 1 Different structures in as-cast and as-hot-rolled specimens (a) a light micrograph of an as-cast state, consisting of large, heterogeneous, and columnar grains, and (b) a thin foil micrograph of an as-hot-rolled state, consisting of fine and homogeneous grains.

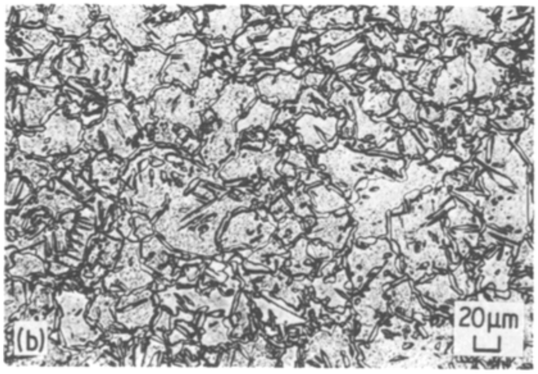
Figure 2 A thin foil micrograph of the specimen quenched from 1100° C, showing a massive martensitic structure.

ties. In the first place, as is shown in Fig. 3, the possibility can be understood by the structural changes arising from heating at a coexisting range. The three kinds of specimens were independently heated to 950° C and followed by iced-brine quenching. Needle-like islands, which correspond to those which have transformed to austenite during heating and then to martensite during quenching, show different shapes, depending on their previous structures. The differences can notably be seen on the morphology and the density of the islands. In the as-cast state (Fig. 3a), the islands are large and heterogeneous; whereas in the other states, (Figs. 3b and c), they are fine and homogeneous. Since the ferritic grains in the as-cast state are large and heterogeneous, as shown in Fig. 1, the resultant islands nucleate along the large columnar grain boundaries, so that the morphology is large and heterogeneous. In the hot-rolled specimens, the islands appear on the recrystallized grain boundaries that were produced during heating at 950° C or during hot rolling, and they are more fine and more uniform, when compared with those of the as-cast state.

On the other hand, the following facts are well known with regard to grain refining: (a) many kinds of boundaries, such as, lath, packet, block boundaries are introduced by the massive martensitic transformation [16], and (b) the lath size in the martensite is proportional to the grain size resulting from the subsequent transformation [17]. For the grain refining, thus, a previous transformation is considered to be the most effective factor. Fig. 3c, for example, which is a structure previously transformed and then heated at 950° C, shows a very fine and homogeneous structure, this being considered to be a reflection of such a martensitic transformation. The thin foil structure corresponding to Fig. 3c is illustrated in Fig. 4, where numerous dislocations are seen in the transformed part and some of the superlattice dislocations in the untransformed part.

From the island morphology, it follows that austenitization which takes place on heating can preferentially initiate on the grain boundaries of ferrite or martensite. Thus, the distribution of the islands is concluded to be determined by that of





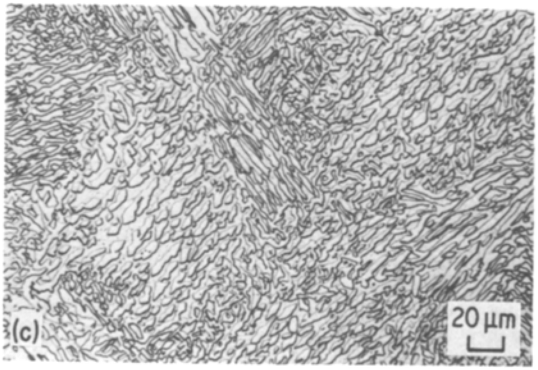


Figure 3 Effect of previous states upon the structural changes occurring in specimens followed by heating at 950° C, where two phases can coexist. (a) as-cast, (b) as-hot-rolled and (c) hot-rolled and then transformed by quenching from 1100° C.

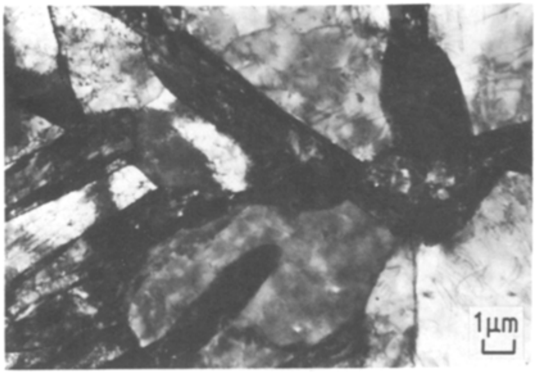


Figure 4 A thin foil micrograph of the specimen transformed and then heated at 950° C for 30 min, showing a mixed structure composed of ferrite and martensite.

grain boundaries in the states preceding the austenitization.

Apart from grain growth, no change in structure is expected to occur during the heating in the temperatures between 925 and 730° C, because only a ferritic phase is stable in this temperature range. Nevertheless, some precipitates were observed in the specimens heated at temperatures from 800 to 900° C for one hour. Fig. 5, for example, shows a thin foil micrograph of the specimen heated at 900° C during one hour, this treatment having caused the most notable precipitation. According to the measurement by analytical electron microscopy, the precipitates could not be identified, although they are composed of vanadium-enriched phases. These phases have been extracted by a conventional replica method and then examined by electron diffraction, but they could not be clarified because of the occurrence of complex lines.

In heating at temperatures below 730° C, the advancement of an order reaction is simultaneously accompanied by the precipitation of the γ phase [3–14, 18–28]; the structural changes have been observed by optical means [14, 20] and by electron microscopy [7, 9, 14, 19, 23, 24, 28]. The γ phase has been regarded as being austenitic, but recently the phase has been found to be enriched with cobalt and vanadium [9, 20]. Consequently, the phase tends to be expressed, rather than as γ, γ' [10, 18, 25, 26] or γ_2 [9, 23, 29]. (hereafter, γ' will be adopted after Köster and Schmid [25, 26] for convenience).

The composition of the γ' phases has been determined by Fiedler and Davis [20], who analysed the precipitates extracted by electrolytically dissolving the matrix aged at 680° C for 48 h, by means of an atomic absorption spectrometer, and conclude it to be: 65%Co–22%V–13%Fe (wt%). The present investigation, on the other hand, has measured directly some of the individual particles of the γ' precipitates, using an analytical electron microscope. The precipitates for the analysis, as shown in Fig. 6, have been extracted from the samples that were annealed at 950° C, cold rolled to 90%, and then aged at 650° C for 16 h; they are concentrated on the particular regions that were austenite at 950° C. The individual points of measurement are shown in Fig. 7. There is some scatter, but the average was concluded to be 64%Co–21%V–15%Fe (wt%). This is in good agreement with Fiedler's result. Ashby *et al.* [9] have suggested that the precipitates can be viewed simply as iron-substituted Co_3V.

Bennett and Pinnel [13] have shown that there

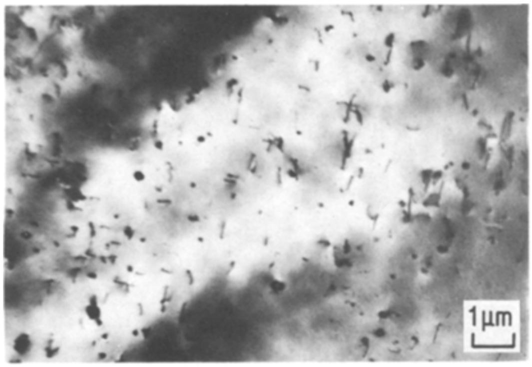


Figure 5 A thin foil micrograph of the specimen annealed at 900° C for 1 h, showing many precipitates which could not be identified.

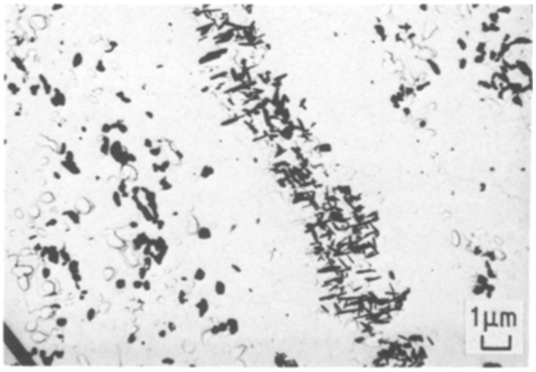


Figure 6 Precipitates extracted from the specimen aged at 650° C for 16 h, after quenching from 950° C and then cold rolling to 90%.

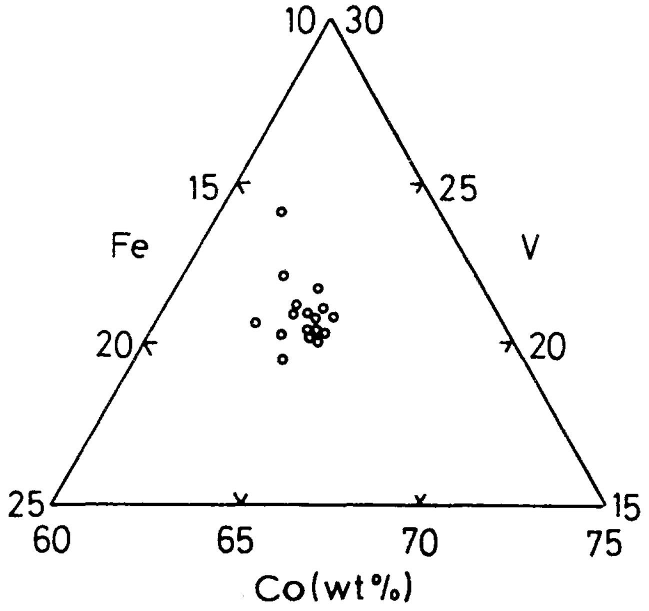


Figure 7 Compositions of the individual particles of the γ' precipitates extracted from the same specimen as that in Fig. 6, showing, on average, to be 64%Co–21%V–15%Fe (wt%).

is a difference in the composition between the martensite, which is austenite at 950° C, and the ferrite; the former is enriched with cobalt and vanadium, more than those in the latter. A similar examination has been carried out for the specimen shown in Fig. 4, using the analytical electron microscope, and it has been revealed that the compositions of the martensite and of the ferrite are: 50%Co–47%Fe–3%V (wt%) and 48%Co–51%Fe–1%V, respectively, over an average of seven points of measurement. These results are similar to that of Bennet and Pinnel [13]. The difference in the composition between the austenite and the ferrite led to a marked difference, at location, in the density of the γ' precipitates, though at room temperature both phases have the same type of lattice, bcc. Fig. 8 shows the microstructure of the specimen heated at 950° C and then aged at 600° C for 5 h, dispersions of plate-like γ' phases being observed in the range from the bottom left to right of the micrograph; the habit plane of the γ' plates have the orientation of $\{1\,1\,0\}\langle 1\,1\,1\rangle$, in complete agreement with other reports [7, 9, 19].

If the properties of the alloys, thus, can be controlled by the distribution of the γ' phases, then care should be taken to ensure that the nucleation of the austenite occurs more uniformly and more densely. Since precipitation is, in general, affected by lattice imperfections, a plastic deformation, if dislocations become the sites, is considered to give a favourable condition for the distribution of the γ'. In Fig. 9 the difference in the distribution of the γ' precipitates is shown for two kinds of different states: the sample in Fig. 9a was as-cast aged at 650° C for 16 h, and (Fig. 9b) the sample was cold rolled to 90% after quenching from 950° C, and then aged at 650° C for 16 h. There is a marked difference in the distribution of the γ': in Fig. 9a the distribution is dilute while in Fig. 9b it is very dense. It follows that a cold rolling is clearly effective for improving the distribution of the precipitates.

3.2. Structures and mechnical properties

The effect of heat treatments on tensile properties is shown for as-cast specimens in Table I. An as-

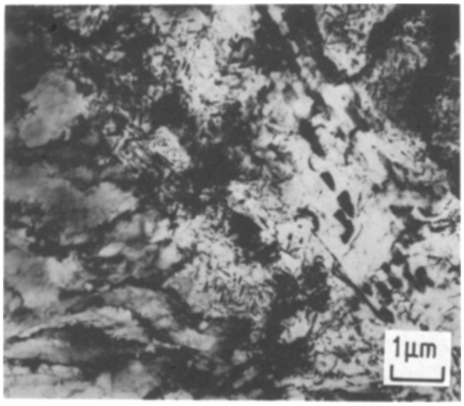


Figure 8 A thin foil micrograph of the specimen annealed at 950° C and then aged at 600° C for 5 h, showing a zone running from the left top to the right bottom of the micrograph in which fine plates of γ' precipitate are observed, while no particles in left bottom corner.

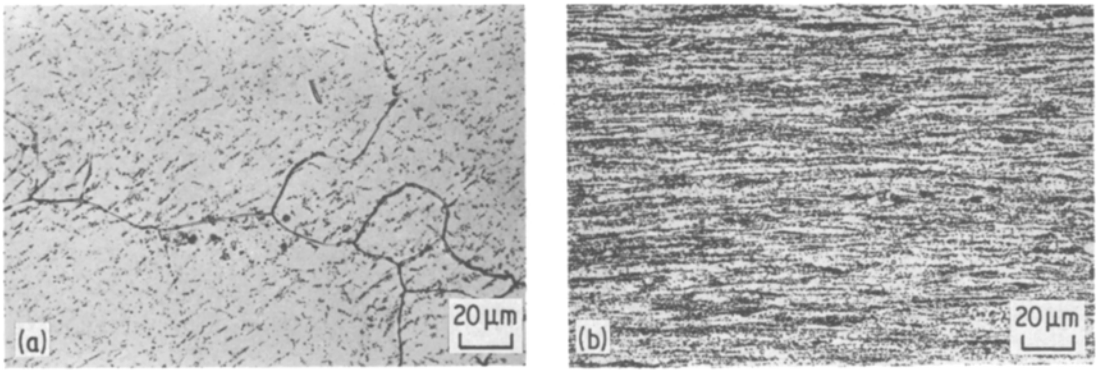


Figure 9 Effect of previous states on distribution of the γ' precipitate in the specimens aged at 650° C for 16 h. In (a) dilute distribution with large particles can be seen for the as-cast specimen, and in (b) dense distribution with fine particles is for the specimen which was quenched from 950° C, cold rolled to 90%, and then aged (longitudinal section).

cast state is very brittle, with no elongation being observed. Quenching from temperatures over 850° C, however, brings about ductility again; the specimens quenched from the ferritic region give the best elongation. The martensitic and mixed structures, which were obtained by quenching from over 1050 and from 950° C, respectively, are inferior to the ferritic structure in elongation. In the as-cast state, the structure showing best elongation is ferritic one which consisted of large and heterogeneous grains; the martensitic structure is not always the best. Ductility is always observed if specimens are only quenched from the ferritic region, irrespective of the morphology of the structures.

An as-cast specimen quenched from 950° C gives only about 7% elongation, whereas the specimen previously transformed and subsequently quenched from 950° C leads to about 20%. Both the phases in the mixed structure, namely, ferrite and martensite, consist, at room temperature, of the same type of lattice, bcc, though there is a small difference in composition. This difference in elongation may probably be attributed to the difference in uniformity and fineness of the islands occurring in the mixed structures, as can be seen in Fig. 3.

Subsequent ordering (carried out at temperatures from 550 to 650° C in the present study) causes the specimens to embrittle, independent of the structural morphology. Even if the most fine and homogeneous structure is obtained by combinations of heat treatments, i.e. transformed and then heated at 950° C, the ordering has caused such a specimen to embrittle. This implies that the occurrence of ductility is produced by quenching, rather than by the refining of grains, and that the grain size factor is a minor one.

Table II exhibits effects of heat treatments for as-hot-rolled specimens. The structures in as-hot-

TABLE I Effect of heat treatments on tensile properties of as-cast specimens

Heat treatments	Mechanical properties	
	Elongation (%)	Tensile strength (kg mm^{-2})
as cast	0 0 0.2	22 22 24
1200° C IBQ	3.5 2.5 6.5	62 69 93
1050° C IBQ	10.0 11.5 14.0	47 57 85
950° C IBQ	5.0 6.0 9.0	65 67 69
900° C IBQ	20.0 20.0	61 66
850° C IBQ	20.0 20.0 25.0	59 71 82
1200° C IBQ, 950° C IBQ	18.5 18.5 21.0	55 74 75
1050° C IBQ, 950° C IBQ	20.0 22.5	57 71
1200° C IBQ, 550° C WQ	2.0 2.0 2.0	59 62 67
950° C IBQ, 550° C WQ	1.0 1.5 1.5	34 40 45
1200° C IBQ, 950° C IBQ, 550° C WQ	1.0 1.5 5.0	46 50 60

Annealing time (over 850° C): 10 min, ageing time (below 550° C): 1 h, IBQ: iced-brine quenching.

TABLE II Effect of heat treatments on tensile properties for as-hot rolled specimens

Heat treatments	Mechanical properties	
	Elongation (%)	Tensile properties (kg mm^{-2})
as-hot-rolled	0	34
1200° C IBQ	12.5	73
1050° C IBQ	13.1	72
900° C IBQ	2.5*	51*
850° C IBQ	4.0*	50*
800° C IBQ	8.5*	75*
1200° C IBQ, 950° C IBQ	18.0	67
1200° C IBQ, 600° C WQ	0	53
1200° C IBQ, 550° C WQ	0	56
1050° C IBQ, 850° C IBQ	20.5	68
1200° C IBQ, 1050° C IBQ, 600° C WQ	1.5	50
1200° C IBQ, 1050° C IBQ, 550° C WQ	1.5	53
1200° C IBQ, 1050° C IBQ, 950° C IBQ‘600° C WQ	1.5	44
1200° C IBQ, 1050° C IBQ, 950° C IBQ, 550° C WQ	1.5	45

Annealing time (over 800° C): 10 min (*: 1 h), ageing time (below 600° C): 1 h, IBQ: iced-brine quenching.

rolled specimens, as in Fig. 1, are very fine and homogeneous, differing with that of the as-cast specimen. However, no elongation is observed in the as-hot-rolled specimens. The specimens subjected to the transformation give about 13% elongation, but those quenched from the temperatures of a ferritic region indicate comparatively less elongations. This phenomena is considered to be related to the precipitation of the unknown phases, as shown in Fig. 5. This expectation may be realized by the fact that heating at temperatures at which such precipitates can dissolve into the solid solution, for example, on heating at 1050° C, causes the elongation to recover to about 20%.

In spite of a wide variation of changes in the structures, the subsequent treatment of ordering allows the specimens to embrittle, independent of the structures resulting from the various heat treatments designed to obtain more fine and more homogeneous structures. Although the fine and homogeneous structure, as is seen in Fig. 3c, can be obtained by a double treatment of transformation, namely, heating at 1200° C and then at 1050° C prior to heating at 950° C, even the specimen thus treated also shows embrittlement after ordering. With the limit of the present study, no improvement in ductility is brought about after ordering, even though the structures were made to be as fine and homogeneous as possible.

4. Discussion

It is well known that the addition of particular elements, such as vanadium or chromium, is effective for improving the ductility of FeCo alloys. Some of the mechanisms for this effectiveness have been put forward. Of these, one has been ascribed to a martensitic transformation by which the grains become increasingly fine and homogeneous [3]. The opinion, however, can be contradicted by the present experiment: (a) the martensitic transformation does not always bring about sufficient ductility, and (b) an as-cast specimen composed of large and heterogeneous grains, even without the transformation by which the grains can be refined, can show sufficient ductility, if only the specimen is quenched from a ferritic region. In addition, the idea that the ductility is caused by the formation of a specified configuration of dislocations [30, 31] can also be denied by the present experiment: even the state in which such a configuration is never made has exhibited ductility, e.g. in the states sufficiently annealed or as-cast, whenever the specimens are quenched from the temperature of a ferritic region. On the other hand, it is the thought that the brittleness in the alloys seems to be associated with any included impurities [3, 32–34]. The previous reports [35, 36], however, have stated in detail with regard to the effect of the impurities on the mechanical properties, and this idea has been contradicted at least for FeCo alloys. The majority of explanations for the brittleness have been concerned with the order–disorder reaction [2, 27, 31–34, 37–46], but the problems have not yet been clarified.

Now that the ductility obtained increases with increasing cooling rate, it may immediately be considered that the order reaction must be responsible for the embrittlement. Although this view would be correct, the following facts have to be explained: the specimens severely cold-rolled, even after ordering, do not always show embrittlement, e.g. in FeCo–V alloys by Pinnel and Bennett [8], in FeCo–C alloys by the author [36], and in FeCo–V alloys in a subsequent paper [15]. Thus, an attempt has been made to interpret such facts, and a proposal, the LCD zone model, has been put forward [35, 36]: if any precipitation takes place, then around the individual particle of the precipitates, a zone in which either cobalt or iron is denuded is occasionally formed: the specimen including such zones would consequently be expected to be able to produce ductility, since these zones are considered to correspond to disordered zones.

According to the model, the precipitate Co_3V should be found. Although the composition of the individual γ' particles has not yet been determined, Fiedler and Davis [20], who have obtained an average value for the individual particles, were the first to reveal the problem. In the present experiment, each of the precipitates has been analysed and they have been proved to consist approximately of Co_3V compound, in which cobalt atoms may partially be substituted with iron ones. In the vicinity of the Co_3V precipitates, therefore, the LCD zones that are previously proposed may naturally be considered to occur.

In this alloy it has been found that the difference in composition between the ferrite and the martensite occurs during heating at the temperature where two phases can coexist, that the portion of the resultant martensite is enriched in cobalt and vanadium atoms, and that the γ' precipitates develop predominantly in the enriched regions, as is shown in Figs. 6 and 8. Assuming that the LCD zones are formed around the individual γ' phases, the mechanical properties may be predicted to differ between the region including and that not including γ' phases, because the former, even after ordering, is able to be relatively ductile as the disordered zones are brought about by the formation of the LCD zones, while the latter is able to be brittle as the disordered zones do not occur or, if any, are present in a small amount. For such a mixed specimen, therefore, the mechanical properties may be governed greatly by the existence of such brittle regions, particularly by the condition of distribution of the brittle phase, even though the ductile regions coexist. In the present experiment, no occurrence of ductility, after ordering, is observed on the specimens heat-treated in such a manner as to be more fine and more homogeneous. This is considered to be attributed to the existence of the brittle phases and to the distribution conditions of the phases. One way to prevent embrittlement resulting from the brittle phases is to increase the volume fraction of the ductile phases and/or to decrease that of the brittle phases, together with a more fine and more dense distribution of both phases. Another way is to allow the specimen to be severely cold rolled. With the rolling, both phases can be elongated along the rolling direction to form a fibrous structure consisting both of ductile and brittle phases. In other words, a fibre-induced effect that is well known in the field of the composite materials, that is, the same effect produced with fibre-reinforced materials, will be obtained. If such an effect is produced, further propagation of the cracks, having arisen from the brittle phases, may markedly be reduced or be prevented, because it should be suppressed by the forest of ductile fibres surrounding the brittle ones. Assuming that the volume fraction and the density of the brittle phases included in a sample are fixed constantly, it is easily considered that the free mean-path of the crack having occurred at a brittle region may be decreased by increasing the reduction of cold rolling, so that a critical reduction may be reached, depending on the volume fraction and the density of the brittle phase. The fact that no ductility has been obtained after ordering may be explained as follows: in the specimens of the present experiment the fibre-induced effect could not be accomplished only by the heat treatments. Since the specimen composed of two phases is a composite but not a fibrous one, the fibre-induced effect does not occur unless the specimen is cold rolled over a critical reduction. It will be shown elsewhere that the critical reduction is about 72% [15].

The hot-rolled plates that are heat treated at and then quenched from the coexisting temperature have been easy to cold roll, with no cracking being observed. This suggests that the dispersed martensite islands have the function of causing the crack propagation to be prevented. In order to increase this function, the following conditions should be satisfied: (a) to increase the volume fraction of the islands, either by increasing the vanadium content or by widely changing the ratio of iron to cobalt, (b) to cause the resultant islands to be fine and dense as much as possible, by the combination of heat treatments and cold rolling; the best distribution may be obtained when the fibre-induced effect is developed, e.g. by a cold rolling.

Although in the discussion mentioned above a fibre-induced effect is restricted in that resulting from the martensite islands, such an effect can be applied to the clusters existing in a solid solution, and this will be shown in detail elsewhere [15].

5. Conclusions

The ductility of the FeCo–2V alloy, if only the alloy is quenched from the temperature of a ferritic region, can be obtained even for the as-cast specimen which is composed of large heterogeneous grains. The martensitic transformation, by which the grains can be refined and homogenized, has not been found to be always effective for improving the ductility, implying that the transformation and its refining effect is of minor importance. On the other hand, even in the states quenched from the temperature where two phases, ferrite and austenite, can coexist, the ductility of the specimens are strongly affected by the uniformity of the structure, showing a tendency to increase with increasing uniformity. Within the limits of the present experiment, the specimens always show embrittlement when they were ordered, even with those that had the most refined and the most homogenized structure. The embrittlement caused by the ordering could not be prevented only by heat treatments.

The compositions of the individual particles of the γ' precipitates accompanied by the ordering have been revealed to be 64%Co–21%V–15%Fe (wt%); to be regarded as an iron-substituted Co_3V compound.

Acknowledgement

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