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Development of Cube on Edge or Cube Textures in Iron Base Alloys

—Si-Fe with Ce addition, and Sn-Fe and Ce-Fe—

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Abstract

In an attempt to deal with phenomena involved in grain growth in cold rolled iron base alloys, crystal growth analogy was applied out and the following results are obtained,

- 1) 3.25% Sn-Fe and 1.25% Ce-Fe alloys in alloy systems with Al-Fe type phase diagram develop cube texture,
- 2) 3.25% Si-Fe alloys with S or Ce addition undergo an enhanced development of cube on edge texture which takes place at the temperatures characterized by the phase diagrams of the additions with iron rather than at the so-called secondary recrystallization temperature inherent to Si-Fe alloy,
- 3) the cube texture is formed in the early stages of the grain growth by the initiation of cube nucleus in the texture,
- 4) the decay in the texture at higher annealing temperatures commonly found in alloys developing cube texture is ascribed to the higher rate of emerging liquid phase and randomly oriented nucleation therein.

I. Introduction

Discussions on the grain growth so far held were carried out based on the grain boundary energy as the main driving force, but this agrees only in a few metals or alloys within a narrow temperature ranges^{1)~3)}. It follows that the influences of other driving forces for growth together with the grain boundary energy may exist even in a range defined as normal grain growth.

One of the present authors has attempted to find consistent explanation of the phenomena regarding the grain growth chiefly of cold rolled Si-Fe, Al-Fe alloys. The respective development of cube on edge and cube texture in these alloys were explained by association with the grain boundary with an interface between a growing crystal and the medium surrounding it in accordance with the general concept of crystal growth⁴⁾.

The consideration mainly consists of the following,

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- 1) Regions of high and low concentrations in additions influencing the phenomena arising in grain growth, may be initially induced in primary recrystallization.
- 2) The potential between these regions can motivate the boundaries to migrate in place and/or in co-operation with the grain boundary energy, and
- 3) When either side of the regions attains a liquidus corresponding to its deviated composition, processes similar to those in crystal growth in a solution may occur: namely enhanced growth of grains, oriented nucleation and growth may be seen.

This consideration may bring forth a rather promotive effect of impurities and additions for growth and thereby explain the mechanisms of behaviors such as the enhancement of growth and texture formation.

Here, alloys other than those used in our previous study⁴⁾, Sn-Fe and Ce-Fe alloys were employed, and detailed mechanisms on the enhanced growth of

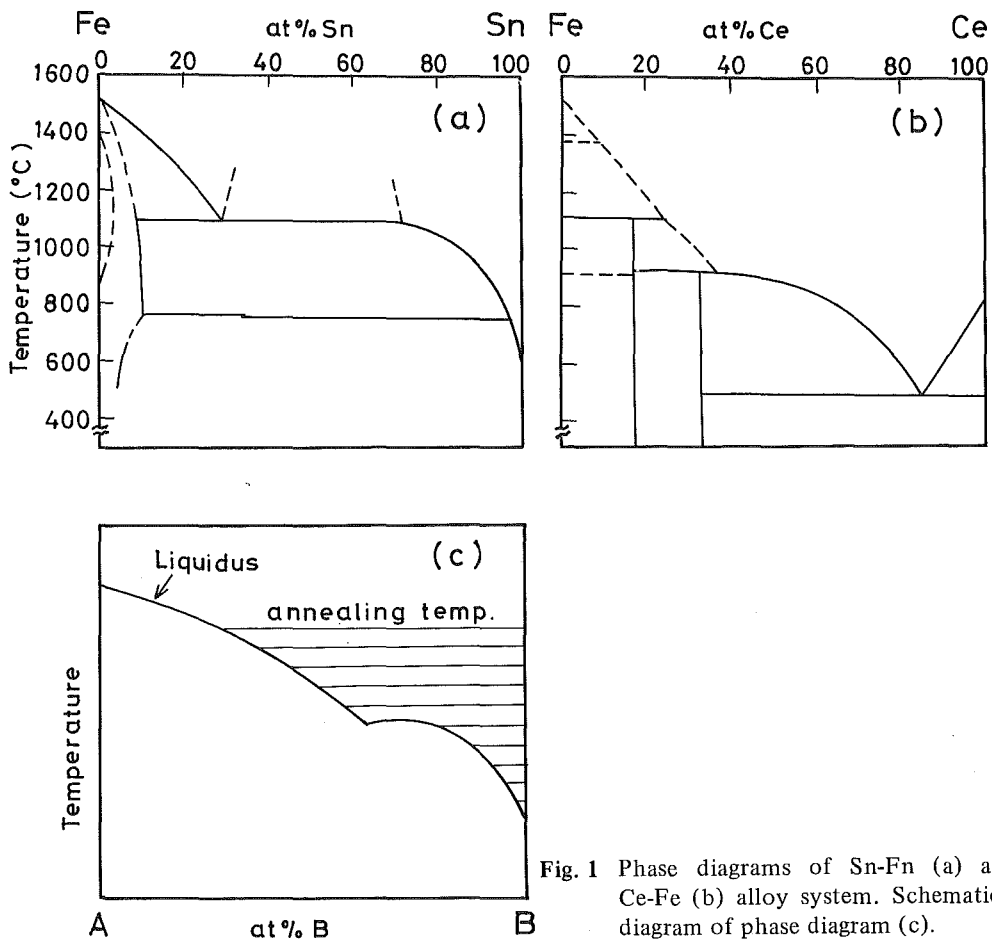


Fig. 1 Phase diagrams of Sn-Fe (a) and Ce-Fe (b) alloy system. Schematic diagram of phase diagram (c).

cube on edge texture and formation of cube texture, and the texture decay at higher annealing temperatures in Al-Fe, Ge-Fe, Sn-Fe and Ce-Fe alloys are presented to confirm the above considerations.

II. Experimental

As the alloys assumed to develop cube texture, Sn-Fe and Ce-Fe alloys were prepared and the alloys, Si-Fe, Si-Fe with a small addition of sulfur and Al-Fe in the previous study⁴⁾ were also used. The methods for preparing the materials and observing the processes were also the same as those adopted in the study mentioned above.

III. Results and discussion

The experiments on the development of cube and cube on edge textures were carried on Sn-Fe and Ce-Fe alloys, Al-Fe and Si-Fe alloys with S and Ce additions respectively.

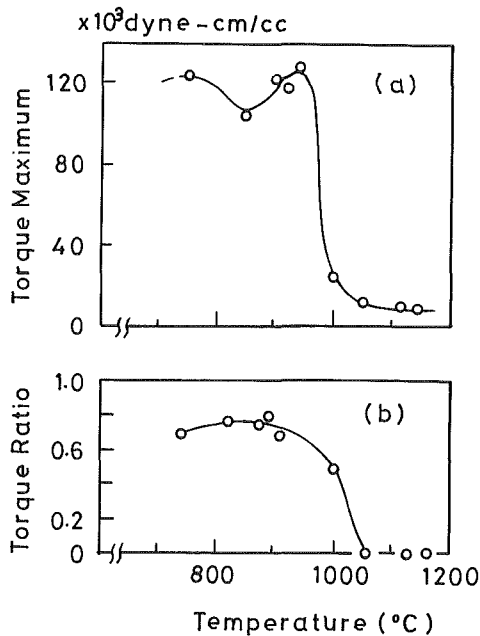


Fig. 2 Torque maximum-temperature (a) and torque ratio-temperature (b) in 3.25% Sn-Fe alloy annealed in a dry hydrogen atmosphere.

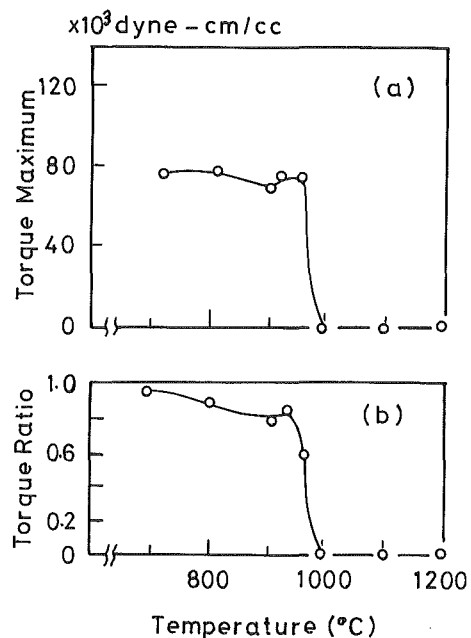


Fig. 3 Torque maximum-temperature (a) and torque ratio-temperature (b) in 1.25% Ce-Fe alloy annealed in a dry hydrogen atmosphere.

1) Grain growth in Sn-Fe and Ce-Fe alloys

The phase diagrams of Sn-Fe and Ce-Fe alloy systems are shown in Fig. 1 (a) and (b), which represent the liquidus descending toward the added elements, Sn or Ce, similar to that in the case of Al-Fe alloy (let it be Al-Fe type). The growth is influenced by the shapes of these liquidus including the characteristic points such as eutectic point, and also area from the liquidus up to annealing temperature as schematically represented in Fig. 1 (c).

The curves of the torque maximum- and torque ratio-temperatures of Sn-Fe and Ce-Fe alloys are shown in Fig. 2 (a) and (b), and Fig. 3 (a) and (b) respectively. These alloys developed a cube texture judging from the fact that the torque ratio was due to shift from 0.43 to unity as the texture shifted from (110) [001] to (100) [001] main component through varying the concentration ratio of these two components⁴⁾.

It may also be noted that the alloys attained highly aligned texture early at lower annealing temperature which may due to the presence of liquid phase.

A drop at about 910°C in the curve was also observed⁴⁾.

2) Grain growth in 3.25% Si-Fe alloy with S and Ce addition

3.25% Si-Fe alloy with 0.021% S addition was reported to show remarkable development of cube on edge texture in excess of 960°C⁴⁾. The specimens annealed at the temperatures corresponding to the plots on the torque curve in Fig. 4 were analysed and the values are given in Table 1.

Table 1. Sulfur content (wt %)

Annealing temperature °C for 1 hr	960	980	1000	1020	1040
S	0.021	0.018	0.023	0.020	0.020

The values of S may presumably be still in excess of the solubility limit in grain boundaries⁵⁾ which is about 0.04 at about these temperature range in Si-Fe alloy⁶⁾ and thereupon the growth may be carried out in the presence of two phase, one of which is due to the liquid phase at these temperatures.

The growth curves represented by torque maximum- and torque ratio-temperatures on 3.25% Si-Fe alloy with 0.10% Ce addition are shown in Fig. 5 (a) and (b). From the rather complicated curve shape, the alloy is shown to develop cube on edge texture at a lower temperature, and then tends to develop cube texture at higher temperatures. These processes were substantially influenced by the shapes of phase diagrams of Si and Ce built with Fe.

The fact that the cube on edge texture developed at about 800°C will

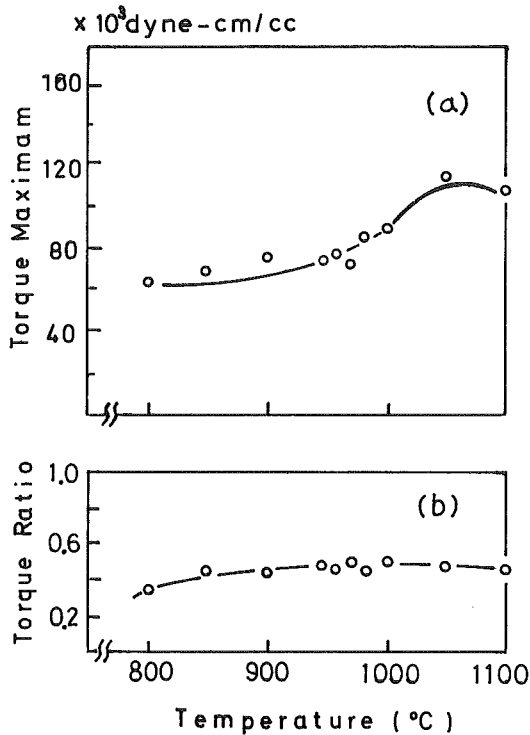


Fig. 4 Torque maximum-temperature (a) and torque ratio-temperature (b) in 3.25% Si-Fe alloy with 0.021% S addition at higher annealing temperature in a dry hydrogen atmosphere.

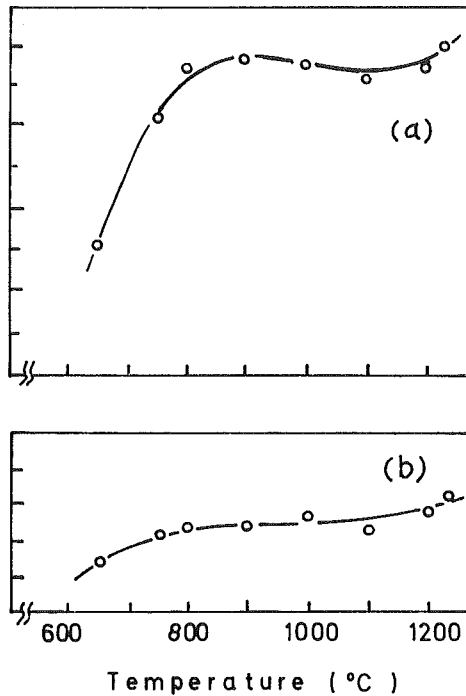


Fig. 5 Torque maximum-temperature (a) and torque ratio-temperature (b) in 3.25% Si-Fe alloy with 0.10% Ce addition in a dry hydrogen atmosphere.

signify that no temperature defined as secondary recrystallization temperature inherent to Si-Fe alloy in excess of about 1000°C existed but that the influence was lead from the character of the phase diagram between the additions and iron as mentioned above.

The somewhat dispersed plots may be attributed to the fact that they were not the temperature sequences on the same specimen in this case.

3) Mechanism of cube nucleation in Al-Fe type alloys

Here, the mechanism of cube nucleation will be considered. The precise plots on the torque maximum- and torque ratio-temperatures in Al-Fe alloy up to 800°C are shown in Fig. 6 (a) and (b). It is seen that the cube texture develops in this temperature range from these curves in particular from the gradient of torque ratio. The optical micrographs in the same range in Photo. 1 (a) reveal that there are many doubled grain boundaries running in parallel with

the rolling direction. This structure is considered to be substantially related with the mechanism of the cube texture formation.

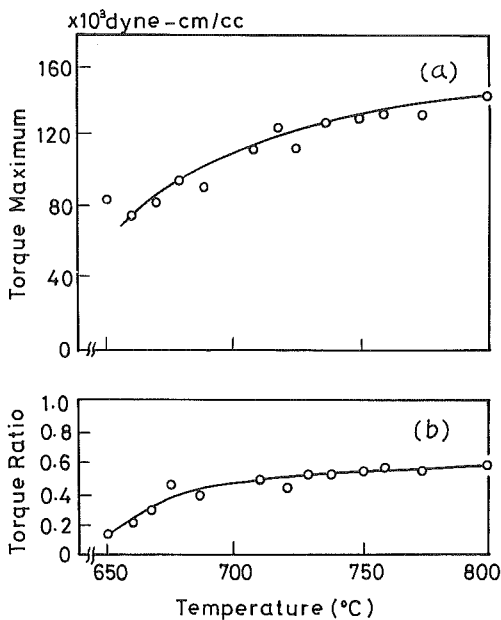


Fig. 6 Torque maximum-temperature (a) and torque ratio-temperature (b) in 3.25% Al-Fe alloy annealed at lower temperature in a dry hydrogen atmosphere.

It is surmised that the grains small and rather elongated in the rolling direction immediately after the primary recrystallization tend to grow in such a way as to take a more stable shape and size at an ascending temperature. This may be achieved by the boundary migration at higher rate which is perpendicular rather than being parallel to the rolling direction. Thus more regions aligned parallelly to the rolling direction may appear. These are compared with those of Si-Fe alloy with S addition annealed at 980°C for 1 hr whose direction disperses rather randomly as seen in Photo. 1 (b).

These regions in Al-Fe alloy may be ones differing in concentration in Al, with higher Al and taking a liquid phase at that annealing temperature. Then, cube texture formation may substantially result in the problem of oriented nucleation and crystallization in a solution^{7)~9)}. Here, the cube grains seem to nucleate in these regions. Then, the direction and the plane of rolling may be most influential factors on determining the orientation of initiating nucleus as to take cube orientation.

4) Decay in textures at higher annealing temperatures

The decay in texture is generally observed at higher annealing temperatures in the alloys employed which developed cube texture. The approximate values

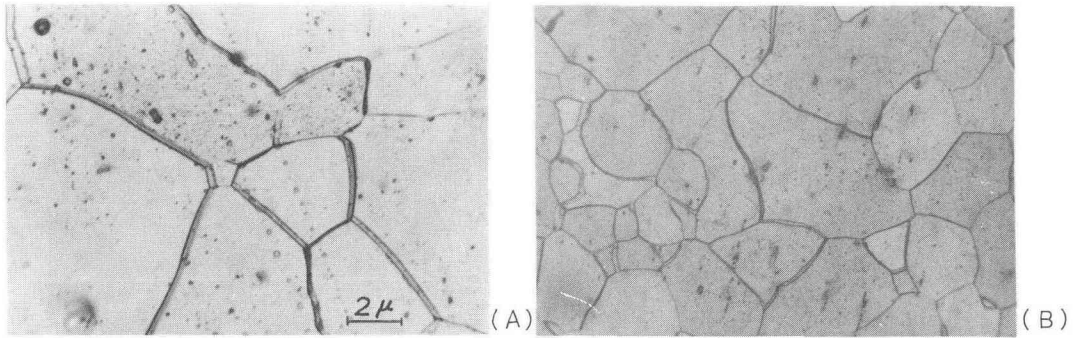


Photo 1 Configuration of aggregates of grains in optical micrograph on 3.25% Al-Fe alloy annealed at 660°C for 1 hr (a) and on 3.25% Si-Fe alloy with 0.021% addition annealed at 980°C for 1 hr (b), both in a dry hydrogen atmosphere.

of the initial and final temperatures of the decay are summarized in Table II.

Table II. Initial and final temperatures of fall in torque maximum curves

Alloy	Al-Fe *	Ge-Fe **	Sn-Fe	Ce-Fe ***
Initial Temp. (°C)	1220	1050	920	910
Final Temp. (°C)	1450	1115	1040	1000

* The value is an extrapolation of the original curve of Bozorth¹⁰⁾.

** The values are from a previous study⁴⁾.

*** Ce-Fe may not be regarded as the same as others, since A₃ transformation is to appear at 910°C.

The temperatures are in the descending order of Al-Fe, Ge-Fe, Sn-Fe and Ce-Fe. For this reason, phenomenon such as penetration of the oxide film from surface may not be adequate, since the effect is so profound. Thus, the area above liquidus up to annealing temperature as shown in Fig. 1 (c) may give some measure for the process. If the emerging rate of the liquid is so high that it can no longer be regarded as approximately equilibrium, the phenomena such as enhanced growth, oriented nucleation and growth may diminish and the randomly oriented nucleation and growth may occur in the liquid. The optical micrographs of some steps in the decay shown in Photo. 2 reveal abnormally grown grains deviated in orientation from (100) [001] and (110) [001] orientations.

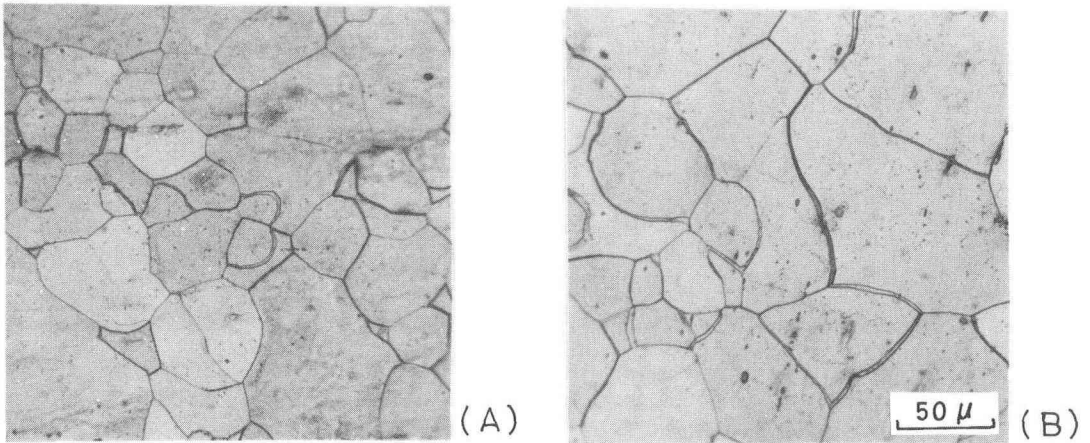


Photo 2 Configuration of aggregates of grains in optical micrograph on 3.25% Ge-Fe alloy annealed at 940 and 980°C for 1 hr in a dry hydrogen atmosphere.

5) *Additional argument*

A considerable portion of the data by the torque magnetometer method employed here had a direct bearing on the conclusion. This method has been utilized for a long time for quantitative measurements because of its convenience in the determination of the textures of iron or iron base alloys, but its reliability is considered to be low in particular in a lower torque value range which corresponds to the range defined as normal grain growth inherent to cold rolled Si-Fe alloy.

The use of the data for the discussion, however, was considered to be quite significant even in meager events, since the values were frequently checked against those of the pole figures and optical micrographs, and were found to be consistent in both arguments to a reasonable extent regarding the numerous specimens used.

IV. Conclusion

Experiments were carried out to clarify the mechanisms involved in the grain growth in cold rolled iron base alloys as a sequence of the previous study.

The development of cube texture in Sn-Fe and Ce-Fe alloys, the detailed mechanism of initiation of cube oriented nucleus in Al-Fe alloy, the enhanced growth of cube on edge texture in Si-Fe alloys with S or Ce additions, and the texture decay in higher annealing temperatures were experimentally confirmed as a sequel to the previous study.

The conclusions may be summarized as:

- 1) Sn-Fe and Ce-Fe alloys also developed cube texture,
- 2) the mechanism of the development of cube texture was explained as an initiation of cube nucleus in a liquid phase aligned in parallel with the rolling direction in an early stage of recrystallization,
- 3) the enhanced development of cube on edge texture in Si-Fe alloy with a small addition of sulfur was reaffirmed to be due to the liquid phase as a second phase by chemical analysis of sulfur in a few points during grain growth, and
- 4) the texture decay at higher annealing temperatures in these iron base alloys was ascribed to the liquid phase with a higher emerging rate than that regarded to be approximately in equilibrium.

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