ELECTRON MICROSCOPIC STUDY OF LAMELLAR EUTECTOID IN A HYPO-EUTECTOID ALUMINUM BRONZE

Thesis for the Degree of M. S.

MICHIGAN STATE UNIVERSITY

C. P. Karnath

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ABSTRACT

ELECTRON MICROSCOPIC STUDY OF LAMELLAR EUTECTOID IN A HYPO-EUTECTOID ALUMINUM BRONZE

By

C. P. Kamath

This work is an investigation of the interface between the f or β_1 -phase and the eutectoid lamellae in a hypo-eutectoid aluminum bronze. It has been found under the electron microscope that both α and γ_2 lamellae touch the β_1 -phase unlike the small gap of ferrite present between the cementite lamellae and the austenite interface in steels as found by Darken and Fisher. The β_1 -rosettes seen under the optical microscope are found under the electron microscope to be clusters of oval shaped masses abutting each other. Again, the dark field, seen under the optical microscope, in between the β_1 -rosettes is found under the electron microscope to be miniature rosettes, perhaps of β_1 , just after nucleation and growth.

ELECTRON MICROSCOPIC STUDY OF LAMELLAR EUTECTOID IN A HYPO-EUTECTOID ALUMINUM BRONZE

Ву

C. P. Kamath

A THESIS

Submitted To
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

Department of Metallurgy, Mechanics and Materials Science

ACKNOWLEDGEMENT

The author is indeed very much indebted to Dr. A. J. Smith for his valuable suggestions, guidance and also for affording all the facilities of the Metallurgy Department for the completion of this work. He is also grateful to Dr. D. E. Scherpereel for his valuable guidance in the electron-micrograph work and having taken the electron microphotographs. Finally he wishes to express his gratitude to the United States Agency for International Development (U.S., A.I.D.) and the Government of Maharashtra for having deputed him for studies and research at Michigan State University.

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I. INTRODUCTION

Aluminum Bronzes i.e. the copper-rich alloys of the Cu-Al system have been the subject of study ever since H. C. H. Carpenter and C. A. (1)

Edwards brought their useful properties before the engineering world. These alloys have tensile strengths ranging from 60,000 to 110,000 psi after different heat treatments. Elongation may vary from 5 to 30% and Brinell hardness number from 120 to 250. These alloys can be rolled or forged and, with some difficulty, are machinable. Their most important and useful property is resistance to corrosion, especially marine corrosion.

Though many investigators carried out work mainly on mechanical properties after quenching and tempering heat treatments, very few have worked on the morphology of the structures of these alloys.

The copper-rich portion of the Cu-Al system is shown in Fig. 1.

The eutectoid decomposition of the β - phase of these alloys is in many respects similar to those of steels both in equilibrium cooled, and quenched conditions, and this fact was recognized at an early date even though the kinetics of decomposition in both the systems have yet to be fully understood. The decomposition of β - phase is somewhat simpler to experiment with than in steels because of lower temperatures involved and also the more sluggish diffusion of the substitutional aluminum atoms than the interstitial carbon atoms in steels. These alloys are amongst some of the non-ferrous alloys used for studying the martensitic-type and other transformations, and carrying the results to experiment with and understand martensitic and other transformations in steels, since most of the non-ferrous alloys can be studied at lower temperatures and hence it is easier to carry out experimental work.

There are some differences in the crystal lattices of these alloys and those of steels. For example, Austenite in steels is F.C.C. whereas, the corresponding phase, β in aluminum bronzes is B.C.C. In the decomposition of this β - phase by quenching, we get meta-stable transition constituent or constituents, β_1 and/or β and hence some complexity of transformation is met with; whereas in the case of steels no such meta-stable transitional constituents have been found as yet and, perhaps, may not be existing.

In the decomposition of austenite of eutectoid composition to its constituents ferrite plus cementite (pearlite), there is controversy as to which of the above two constituents, ferrite or cementite is the initial nucleating agent. Most of the investigators are in favour of cementite to be the nucleating agent of pearlite, but there are others who support ferrite to be the one nucleating first, at least in hypoeutectoid steels. The compromising formulas have also been propounded.

Recently L. S. Darken and R. M. Fisher (4) have used the electron microscope to get much higher magnifications than it is possible in optical microscopes, to get better resolution of the tri-junction of austenite, ferrite and cementite. Their finding of a small gap of, perhaps, ferrite between the austenite interface and the cementite lamellae is an important contribution towards the understanding of pearlite formation.

Since aluminum bronzes also show a lamellar eutectoid and other types of eutectoids, which are called by some authors pearlite **, con-

^{*} There is controversy regarding the existence of β "(3).

^{**} Now-a-days, the term pearlite is used in systems other than ironcarbon system to denote eutectoid transformation products in metallographically analogous structures.

sisting of $\alpha_{\rm cu}$ and γ_2^* constituents in alternate layers as a result of the decomposition of β - phase, the electron microscope can be used to study the morphology of it. The study in this work has been carried out by this method.

^{*} Before 1948 γ_2 was designated by the symbol δ .

II. PREVIOUS WORK AND BACKGROUND

Early investigators like H. C. H. Carpenter and C. A. Edwards (1), B. E. Curry (5), J. M. Greenwood (6), P. Braesco (7), J. Bouldoires (8), Matsuda (9,10), C. Grard (11), I. Obinata (12) and others mainly studied mechanical properties, changes in length, electrical conductivity, thermo-electricity after quenching and tempering at different temperatures or studying after different cooling rates.

It was Matsuda who, on the basis of study of electrical conductivity during cooling at the rate of 10°C/min. , observed decrease in electrical resistance and in length instead of the usual increase during slow cooling rates. This he attributed to the existence of a transitional phase, β_1^* being formed with electrical resistance and volume less than those of β - phase and $\alpha + \delta$ **. I. Obinata (12) confirmed this on finding double breaks in electrical conductivity curves at the eutectoid temperature i.e. $\beta \Longrightarrow \beta_1 \Longrightarrow \alpha + \delta$ even under equilibrium conditions. This he confused with the quenched martensite product β^1 *** whose structure he determined to be hexagonal. Obinata's finding of a double break in the electrical conductivity curve has not been confirmed by other investigators.

The structure of β_1 has been determined to be ordered B.C.C. by G. Wasserman⁽¹⁴⁾ while that of β to be disordered B.C.C.⁽¹⁵⁾ and this was later confirmed by E. Kaminsky et al⁽¹⁶⁾ and by E. P. Klier and S. M. Grymko .

^{*} In earlier works, β_1 was designated with the symbol β^1 ; but, nowadays, β^1 is used to denote martensite containing up to 13.2 % Al (3).

^{**} Since 1948, δ has been renamed as γ 2.

^{***} This product has been previously called acicular eta and a 1. (13)

- E. S. Davenport and E. C. Bain (18) developed the technique of isothermal transformation (T-T-T curves) for studying eutectoidal and other decompositions; and this technique was widely used for studying steels. In other fields, it was less applied.
- C. S. Smith and W. E. Lindlief⁽¹⁹⁾ applied isothermal transformation technique for a detailed study of the decomposition of β phase in an aluminum bronze containing 11.87 % Al and produced outstanding work. They did not concur with I. Obinata⁽¹²⁾ to a large extent and definitely concluded that β_1^* was a transitional meta-stable constituent and not to be confused with the martensite β^1 **. Their theory is that, preeutectoid α nucleates from β and forms a needlelike structure. β_1 is richer in aluminum content than β . With the disappearance of β , eutectoid formed and when once this formed, it grew indiscriminately in both β_1 and β giving rise to difference in tints according to whether the eutectoid formed from β_1 or β . The mode of formation of eutectoid and the primary nucleating agent has not been made clear.

The next elaborate investigation, by isothermal transformation method, was by D. J. $\text{Mack}^{(13)}$ who worked on aluminum bronze containing 11.8% Al; and he confirmed most of the results of C. S. Smith and W. E. Lindlief. According to him, both precutectoid a and a ***, with a predominating, are rejected prior to cutectoid reaction. The cutectoid reaction is "by rejecting a and/or a."

E. P. Klier and S. M. Grymko (17) investigated alloys containing 11.9 and 13.5 % Al by isothermal transformation method and in addition

^{*} Indicated as β^1 in C. S. Smith and W. E. Lindlief's (19) work.

This martensite has been, previously, called acicular β .

^{***} Since 1948 \upbeta has been renamed \upgamma_{2} .

studied the x-ray diffraction patterns of the transformations produced. They also confirmed previous investigators' results and, in addition, postulated the existence of another transitional metastable constituent β'' which is copper-rich and formed in the temperature range 515 - 450°C and perhaps lower. This β'' is thought by them to be the same as the β' (present β_1) found by C. S. Smith and W. E. Lindlief. The discrepency in their assumption is that they say β'' is "aluminum dilute" whereas Smith and Lindlief call their β' aluminum rich. However, the existence of β'' has been disputed by C. W. Spencer and D. J. Mack (3) and in their opinion both β_1 and β'' are the same phase. E. P. Klier and S. M. Grymko concluded that, close to eutectoid composition, β_1 decomposes mainly to α before γ_2 forms, whereas, away from eutectoid composition i.e. at 13.5% aluminum, γ_2 precedes α in the decomposition of β_1 .

R. Haynes has investigated three alloys having the following compositions:

- (a) 11.40% Al and Nil Ni,
- (b) 10.57% Al and 1.02% Ni,
- (c) 10.54% Al and 3.03% Ni.

The method used was by isothermal transformation. He also studied the effect of the presence of nickel. In alloy (a) he did not come across β_1 phase but came across very little lamellar pearlite, the others having none. He concluded that after the formation of proeutectoid α , due to concentration gradients in β , γ_2 nucleates as a sheath around α and between α and β . Next both α and γ_2 grow into β .

In a later investigation, R. Haynes (21) investigated four alloys containing more than twelve per cent aluminum and various percentages of nickel.

The eutectoid composition of the β -phase is 11.8% aluminum ^(22,2). Only R. Haynes has investigated hypo-eutectoid alloys in addition to hyper-eutectoid ones whereas all others have investigated hyper-eutectoid alloys only. The information given by R. Haynes on the nickel-free hypo-eutectoid alloy is meagre; and much of the information is on hyper-eutectoid alloys and that too in the presence of nickel.

Eutectoid transformations, similar to the one in aluminum bronzes, are found in other alloys, the most important and widely investigated one being that of iron-carbon system. In spite of enormous investigations being done, iron-carbon eutectoid decomposition is yet to be fully understood even qualitatively. The inherent difficulties, due to numerous variables and complexities of transformation for its full appraisal, have been responsible for the enormous work done on it as iron-carbon alloys are quite important ones.

Though F. C. Hull and R. F. Mehl's (23) theory of formation of lamellar pearlite from austenite with cementite as the initiating nucleating agent has been widely accepted, yet, there are others (24,25,26) who do not share the same view, especially in the case of hypo-eutectoid steels wherein ferrite has been thought to be the initial nucleating agent. A. Dube (27) and H. I. Aaronson(28) have proposed a compromising view stating that ferrite may be the first "informal" nucleus of pearlite formation. However, these various views have not clarified the nucleation mechanism beyond doubt.

Recently, M. Hillert (29) has ably propounded a theory, in which he explains the initial formation of cementite which grows along the grain boundary and forms almost a complete envelope around the grain. Due to this, carbon is depleted in the austenite in this vicinity and ferrite starts forming. In this way, alternately, cementite and ferrite

start competing with each other to form but at a later stage both help each other for their simultaneous formation and lamellar pearlite is obtained.

But the most useful forward step in this field is that of L. S. Darken and R. M Fisher (4). With the help of electron microscopes, they have studied lamellar pearlite formation in different types of steels and have discovered at magnifications ranging from 20,000 X to 36,000 X, a small gap between the cementite lamellae and the austenite interface under different conditions. No such gap exists between the ferrite lamellae and the austenite interface. This gap is up to 0.35 microns in width, the average width being 0.15 microns and they believe that the gap material to be ferrite. Under optical microscopes, these gaps are not at all detectable due to the limiting resolving power of them.

III. SCOPE OF INVESTIGATION

In the previous section, the comparison in the decomposition of the β - phase in aluminum bronzes and the decomposition of austenite in steels has been stated; and the similarities between the two decompositions or transformations are many, even though the kinetics of transformation may differ.

The finding of the gap of ferrite between the cementite lamellae and the austenite interface $^{(4)}$ is significant. No such investigation has been done to find whether there is a gap between the α or γ_2 lamellae and the β or β_1 interface in the transformation of eutectoid in aluminum bronzes. The question is, will there be similarity between the two transformations in this respect also? If not, what is the nature of the variation? To verify this point, lamellar eutectoid of a hypo-eutectoid aluminum bronze has been taken up for study.

Lamellar eutectoid in aluminum bronzes forms only within a fairly narrow composition range i.e. 10.5 to 13.5% wt. aluminum $^{(30)}$ and that too it forms in smaller quantities. Slow cooling is necessary for their proper formation. Large amounts of acicular and cellular eutectoids along with thick alternate irregular layers of α and γ_2 are formed. Outside this composition range the eutectoid is mostly cellular. It is difficult to get lamellar eutectoid from a phase having considerable concentration gradients $^{(20)}$. The farther the over-all composition of the alloy is from the eutectoid, the slower should be the cooling rate to get a lamellar structure.

Therefore, the slower cooling method worked by C. S. Smith and W. E. Lindlief⁽¹⁹⁾ has been used to get lamellar eutectoid. Isothermal transformation technique has not been used. Different cooling rates

were tried so as to get an interface between β and β_1 and the lamellar eutectoid.

Both optical microscopic and electron microscopic structures have been studied for comparison.

IV. MATERIALS AND METHODS

The hypo-eutectoid aluminum bronze used in this study was prepared by melting high purity copper and aluminum in a plumbago crucible under graphite in an electric resistance furnace. It was cast into an ingot, $7" \times 1\frac{1}{2}" \times 1"$, in a metal mould, the pouring temperature being about 1250° C. To get homogenous alloy, the case ingot was remelted at 1250° C under similar conditions as given above and chill cast.

The cast ingot was homogenized at 900°C for 15 hours and next rolled from 1" to $\frac{1}{2}$ " thickness with intermediate reheatings to 900°C . After the rolling operation was over, the ingot was furnace cooled from 900°C . It analyzed:

<u>Element</u>	<u>_%_</u>
Copper	88.10
Aluminum	11.70
Iron	0.10
*Silicon	0.01
*Manganese	0.005
*Nickel	0.005
*Tin	0.005
*Zinc	0.005
*Lead	0.005

^{*} Semi-qualitative

Sample pieces, 1" \times 5/8" \times 1/2" were cut from the rolled ingot for heat treatment at different rates of cooling.

Sample I was heated to 800° C for two hours, after which it was slowly cooled in the furnace to 650° C in about four hours. The cooling, through the critical range, was done at a uniform rate of about 2° C/min. as suggested by C. S. Smith and W. E. Lindlief (19) in order to get rosettes of β_1 and the lamellar eutectoid growing in them.

Therefore, from 650° to 500°C the cooling was done more or less uniformly in about one hour and fifteen minutes. At 500°C, the sample was quenched in water.

The quenched specimen was cut into two equal halves and the newly cut surface of one of the pieces was filed, first polished with emery and finally with fine alumina on the polishing wheel. Etching was done with Buhler electrolytic etching apparatus for three seconds, using 1% aqueous chromic acid solution as etching reagent as suggested by W. C. Coons and D. J. Blickwede (31).

Three more samples were subjected to heat treatments similar to that given to Sample I, the difference being the cooling rates through the critical range were 1° C, $\frac{1}{2}^{\circ}$ C and $\frac{1}{4}^{\circ}$ C per min. respectively. These samples also, after heat-treatment, were polished and etched like Sample I.

Since, Sample I gave the desired results, it was taken up for study. There were a few good junctions between β , β_1 and a few patches of lamellar eutectoid. Other samples did not show the presence of β or β_1 and hence were not taken for further investigation.

Sample I was repolished, lightly etched (this etching was not satisfactory for optical microscope work) and a negative plastic replica was taken using collodin solution supplied by Hitachi Co. along with their 11A electron microscope. The replica was taken on 200 mesh nickel grids and covered with a drop of polystyrene latex solution in water for shadow contrast. The latex solution was dried in a vacuum desiccator. The dried replica was platinum shadowed in a high vacuum unit.

The replica was next studied under the electron-microscope and photographs were taken at 6,000 and 15,000 magnifications. The negatives were enlarged about 4.75 times to get high magnification prints.

Optical microscope photographs of Sample I were taken.

Sample V is a piece of the rolled ingot which was heated to 800°C and slowly furnace cooled in order to have a fully annealed structure for comparison. This sample was also polished and etched with 1% aqueous chromic acid solution as etching reagent. Optical photograph of the structure obtained was taken.

V. RESULTS AND OBSERVATIONS

The aluminum bronze used contained about 11.70% aluminum and hence, the composition is fairly close to eutectoid composition on the hypoeutectoid side. The homogenized and fully annealed sample showed little pro-eutectoid a areas dispersed throughout the sample and their sizes varied. Those which were found in cellular eutectoid regions were smaller in size and more numerous, whereas, acicular eutectoid regions showed slightly larger in size but fewer in numbers. The matrix was mostly consisted of acicular eutectoid, cellular eutectoid and regions of thick alternate layers of a and γ_2 (see Figs. 2 and 3).

Sample I gave the desired results, i.e., β or β_1 interface and lamellar eutectoid junctions. Most of the eutectoid was either cellular or acicular. Only in a few regions, lamellar eutectoid could be found either abutting acicular eutectoid or within the regions of it (see Figs. 4 and 5). The locations of the lamellar eutectoid were fewer and far between. Out of these fewer locations also, the clear junctions between the β or β_1 and lamellar eutectoid were meagre. No Widmanstatten structure was found.

Samples II to IV did not show any $\,\beta$ or $\,\beta_1$ and hence, were not investigated further.

The electron micrographs of Sample I are shown in Figures 6 to 9. Even at 70,000 X, the α and γ_2 lamellae are touching the β_1 interface and look continuous with it, unlike the result found by L. S. Darken and R. M. Fisher in steels. So. the kinetics of formation of lamellar eutectoid, is perhaps, different from that of lamellar pearlite in steels. E. P. Kliers and S. M. Grymko (17) have stated on the basis of finding inter-lamellar space first decreasing with

decrease in temperature of formation and later on increasing with further decrease in formation temperature, that the kinetics of formation of lamellar eutectoid in aluminum bronze is different from that of pearlite formation in steels. The electron-micrograph finding in the present study is a further proof of their statement.

It is probable that both a and γ_2 are forming at the same time. Their growth in eta_1 is more or less well defined.

R. Haynes' (20) work on 11.4% aluminum alloy does not show any formation of rosettes of β_1 . In the present work beautiful rosettes of β_1 have been obtained (see Figs. 2 and 3). In the optical microscope, at higher magnifications, in a deeply etched structure of Sample I, some faint irregular structure was visible in the etched portion between the rosettes of β_1 .

In the electron micrographs, the rosettes are seen to be, actually, roughly oval shaped masses abutting each other in clusters (see Fig. 4 and 6) and this fact could not be seen clearly in the optical micrographs.

The region in between the rosettes (mentioned earlier about the seeing of faint structure under the optical microscope) showed clearly in the electron micrograph to be miniature rosette-like structures (see Fig. 6) which at 70,000 X (See Fig. 9) is clearly indicated to consist of irregular shaped curved masses, perhaps, consisting of oval shaped masses closely abutting each other (not very clear in Fig. 9). It is possible that these miniature masses are actually β_1 just after mucleation and growth. There is indication in Fig. 6 that these miniature masses coalesce and form quite large oval shaped masses which cluster together and are seen as rosettes under the optical microscope.

The large difference in sizes between the big β_1 rosettes and the miniature rosettes and the numerous indistinct etching lines amongst the latter makes them look darker as a whole under the optical microscope when compared with the bigger β_1 rosettes. This is not the case in the electron micrograph and the etching lines are clearly seen because of better resolution.

The mass behind the interface is seen to be a continuous one, like a band, and is β_1 (see Fig. 9). But in Fig. 4 this fact cannot be realized and it looks as if lamellar pearlite is forming indiscriminately both in rosette β_1 and the so-called $\beta^{(19)}$ (dark etched areas) which is actually β_1 under formation. On close examination, it is found that the interface band does not have etching lines crossing it. The miniature rosettes have all coalesced just before the interface.

The complete enveloping of proeutectoid a by rosettes of β_1 has been observed.

The very dark round spots on the electron micrographs are not a part of the structure but they are globules (about 0.26 microns in dia.) of polystyrene latex, dispersed over the replica to give shadow contrast, magnified.

VI. DISCUSSION AND CONCLUSIONS

A. Discussion:

The similarity between the β - phase decomposition in aluminum bronzes and the austenite in Fe-C system has been referred to in Sections II and III. Both β -phase and austenite are solid solutions having eutectoid phase transformation in them.

Austenites of hypo-eutectoid compositions, under equilibrium conditions, decompose into pro-eutectoid ferrite and pearlite consisting of eutectoid ferrite and eutectoid cementite. The same is the case with β -phases of hypo-eutectoid compositions under equilibrium conditions. They decompose into proeutectoid α and eutectoid consisting of eutectoid α and eutectoid γ_2 . Under non-equilibrium conditions, i.e., under different rates of cooling, in Fe-C system, martensite, bainite and pearlite are met with, whereas in aluminum bronzes, we come across β ' and γ ' martensites β_1 -rosettes, acicular, cellular and lamellar eutectoids.

The most recent divergent views on pearlite formation in Fe-C system are those of M. Hillert (29) on one side and L. S. Darken and R. M. Fisher (30) on the other and this has already been referred to at the end of Section II. Hillert argued that the ferrite gap found by Darken and Fisher is due to the rate of quenching which affects the s/so ratio (where s' is the actual spacing and so is the minimum spacing between the lamellae) giving rise to gap; and he gave in support of his argument certain micrographs, wherin with progressive increased rate of cooling, the development of the gap between the cementite lamellae and the austenite interface was shown. Fisher was not satisfied with Hillert's arguments and proposed further experiments to clarify at

least some of the points which Hillert had raised against their paper. However, as Hillert said, the suggestion by C. Benedicks (32) that both ferrite and cementite could act as germs for formation of pearlite, cannot be lightly brushed aside. Though, Hillert's theory of active partnership between ferrite and cementite in lamellar pearlite formation is logical, it has yet to be put to rigorous tests.

In the case of aluminum bronzes enormous work, as in the case of steels, has not been done. The nucleus for the formation of lamellar eutectoid is still to be understood, just like lamellar pearlite in the Fe-C system. The characteristic intervening β_1 -phase complicates a little the understanding of the transformation. The assumption by earlier workers of changes in the composition of α -phase, mostly on the super-saturation side, to account for the decomposition mechanism of the β -phase, under isothermal transformation, has been proved to be incorrect by R. Haynes (33) on the basis of finding, by x-ray method, that the lattice parameter of α did not change during transformation between 560 and 400° C and was in equilibrium with γ_2 (just as ferrite composition is almost uniform in the Fe-C system during transformation).

On the whole both aluminum bronze and the Fe-C systems are close in their transformation mechanisms and morphology. The non-finding of the gap between the α and γ_2 lamellae and the β_1 interface in aluminum bronzes is more in line with that part of Hillert's theory in which he states that there is active partnership between ferrite and cementite in the lamellar pearlite formation, than those of Darken and Fisher, if the analogy between the two systems is accepted and the difference between the experimental methods followed by Darken and Fisher (isothermal transformation) and in the present study (predetermined cooling rate up to a particular temperature followed by quenching) are taken

into consideration. From a close examination of Fig. 7, it is clear that the β_1 interface is continuous throughout both where the α and γ_2 lamellae are touching it and this means that the transformation is continuous at the interface.

B. Conclusions:

Just as in eutectoid and hyper-eutectoid aluminum bronzes, in hypo-eutectoid ones also rosette shaped eta_1 phase is met with.

The dark matrix seen under the optical microscope between the rosettes of β_1 is found under electron microscope to be made up of miniature rosettes, which, perhaps, are also β_1 phase. The difference seen under the optical microscope between the dark matrix and the bright colored rosettes of β_1 is due to the large difference in the sizes of the two types of rosettes and the numerous etched boundaries of the miniature rosettes.

The big rosettes are clusters of roughly oval shaped masses abutting each other in groups, the abutting lines being not clear under the optical microscope. These have grown out of the miniature rosette masses coalescing together.

The miniature rosettes also consist of roughly rounded masses.

The phase next to the interface where the lamellar eutectoid is growing into, other than big rosettes of β_1 , is a continuous band of coalesced miniature rosettes.

Very little lamellar eutectoid is met with and that too, they are found inside the regions of acicular eutectoid or at the junctions of acicular eutectoid and the untransformed β_1 .

The lamellae of both a and γ_2 touch the interface of β_1 (70,000 X). It is clear from Fig. 7 that the β_1 interface is continuous

throughout, both where the lpha and γ_2 lamellae are touching it.

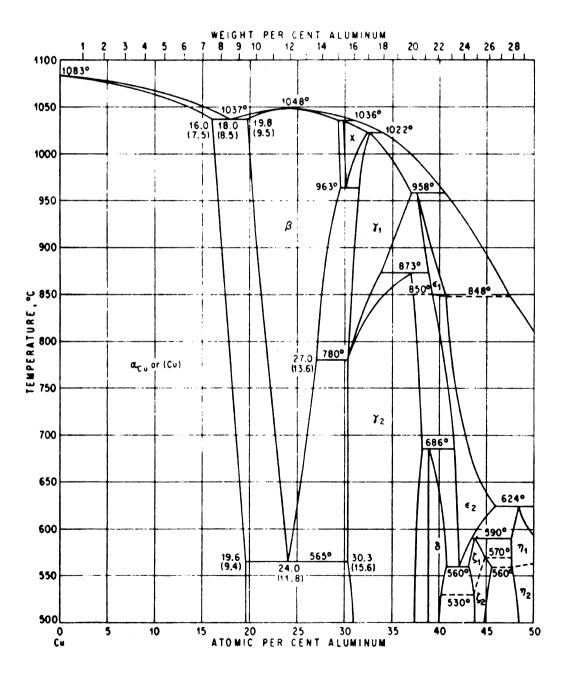


Fig. 1 Copper-rich portion of the copper-aluminum equilibrium diagram.

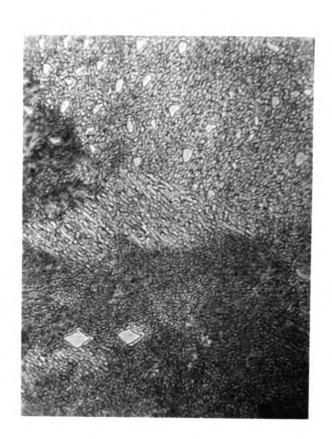


Fig. 2 Fully annealed sample. 250 X.

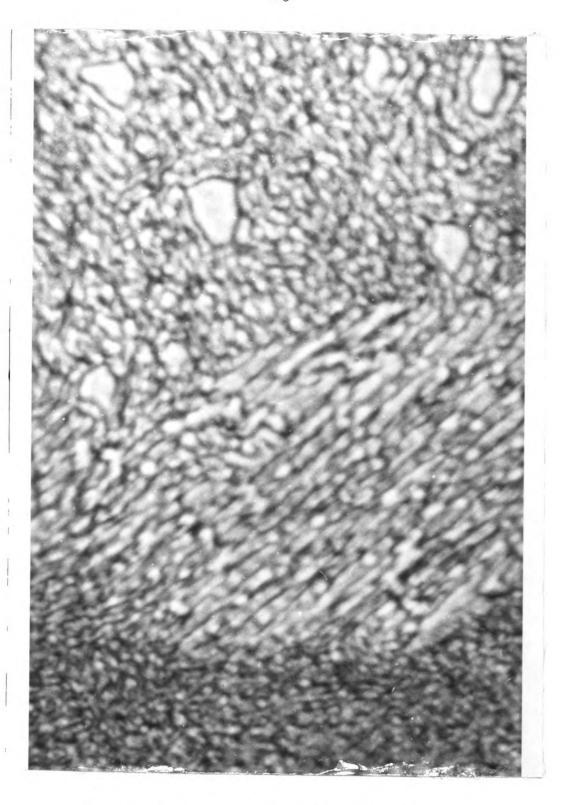


Fig. 3 Fully annealed sample. 1200 X.

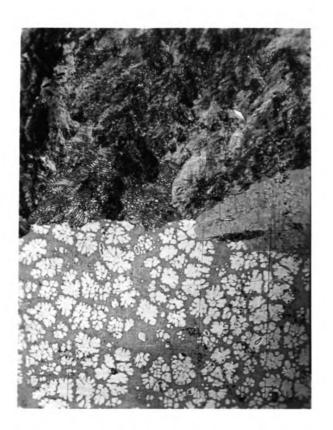


Fig. 4 Sample cooled at 2°C/min. between 650° and 500°C and quenched at 500°C. 250 X.

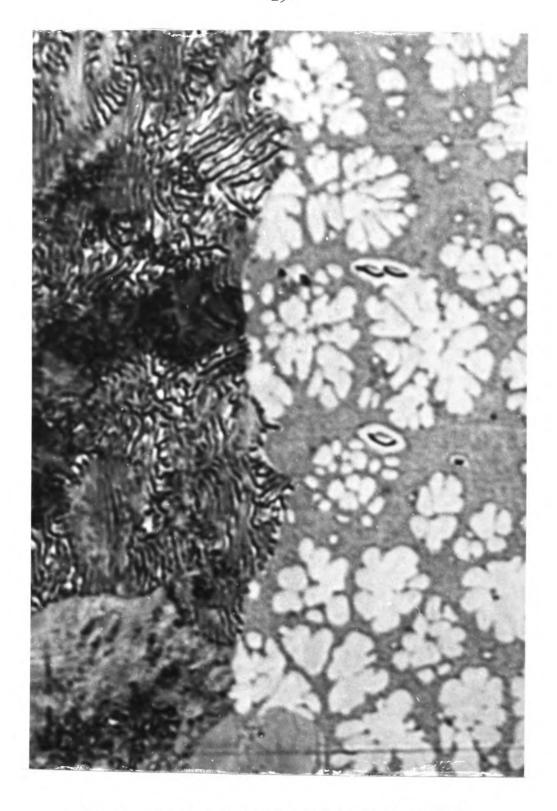


Fig. 5 Sample cooled at 2°C/min. between 650° and 500°C and quenched at 500°C. 1200 X.

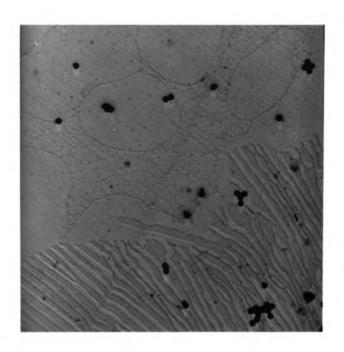


Fig. 6 Electron micrograph of the sample in Fig. 4. 6000 X. (Plastic replica)



Fig. 7 Electron micrograph of the sample in Fig. 4. 28,000 X (Plastic replica)

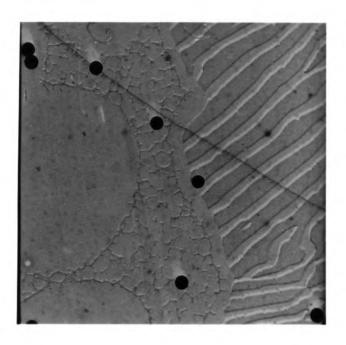


Fig. 8 Electron micrograph of the sample of Fig. 6 in a different spot. 15,000 X (Plastic replica)

Fig. 9 Electron micrograph of the sample of Fig. 6 in a different spot. 70,000 X (Plastic replica)



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