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RESEARCH ON THE FREEZING AND MELTING POINTS
OF IRON ALLOYS

W. Hume -Rothery

Isaac Wolfson Professor of Metallurgy

University of Oxford
Oxford, England

TECHNICAL REPORT

CONTRACT NR. AF 61(514)-1062

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ASTIA

RESEARCH ON THE FREEZING AND MELTING POINTS
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Technical Report
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I.

At room temperatures the metal iron exists in the α - (body-centred cubic) modification and, on raising the temperature, this structure persists until 910°C . (the A_3 point) above which the face-centred cubic or γ -modification is stable until 1390°C . (the A_4 point) where the structure changes back to that of the δ - (body-centred cubic) form, and this modification persists until the melting point (1535°C). When iron is alloyed with a second metal, all the above transformation temperatures are altered and, in accordance with the Phase Rule, the transformations, which take place at constant temperature in pure iron, occur over ranges of temperature in the alloys. The object of the research work described in the present report was to examine the effects of solute elements, mainly from the Second and Third Transition Series, upon the A_4 point and melting point of iron, and in this way to establish the iron-rich portions of the equilibrium diagrams at high temperatures. A further object of the work was to determine the simultaneous effect of carbon and nickel, and for this purpose it was necessary to redetermine the iron-rich part of the equilibrium diagram of the system iron-carbon.

II.

In binary systems where solid solutions are formed, the equilibrium diagrams of iron alloys fall into two main classes:

(a) Ferrite Stabilisers

Solutes of this type have the effect of lowering the A_4 point and raising the A_3 point and, in systems where the solid solutions are of sufficiently wide extent, the phase boundaries join up to form a ∇ -loop, as shown in Figs.1(a) and 1(b). In some systems the A_4 point may be lowered, and the A_3 point raised, without the formation of a ∇ -loop, and the details of the diagrams vary from one system to another. Manganese behaves abnormally but, as regards the other transition metals, ferrite stabilisers belong to Groups IVA, VA, VIA, and VIIA, i.e. to the Groups to the left of iron, when the Periodic Table is drawn in its usual form. The present work has confirmed this principle for the effect of Tc on the A_4 point, and has shown that Ga, like Al, gives rise to an equilibrium diagram of the ∇ -loop type.

In the general case, the formation of a solid solution involves lattice strain, and this is more serious for the solid than for the liquid solution. The liquid phase is, thus, favoured and the liquidus and solidus curves fall as in Fig.1(a). If, however, the second element is of much higher melting point than iron, its latent heat of fusion* and interatomic bonding forces are greater, and these characteristics may be carried over into the solid solution with the result that, after a certain concentration is reached, the solid solution is increasingly stabilised with respect to the liquid, and the liquidus and solidus curves pass through a minimum point, as in Fig.1(b). Examples of this

*To a first approximation the entropies of fusion $\Delta H/T_m$ of all metals are constant, so that the latent heat of fusion ΔH , increases with the melting point, T_m .

are shown by the system Fe-Mo and Fe-W, and to a lesser degree Fe-V and Fe-Cr (see Fig.8). We shall use the term M.P. effect to describe this effect in which the liquidus and solidus tend to be raised if the melting point of the solute is much greater than that of the solvent, or to be lowered if the solute has a much lower melting point. This effect is discussed in greater detail in Section V below.

(b) Austenite Stabilisers

With solute elements of this class, the A_4 point is raised, and the A_3 point is lowered. Since the general tendency is for the liquidus and solidus curves to fall, the raising of the A_4 point means that the δ/γ solubility curves rise to meet the liquidus and solidus, and a peritectic reaction results, as shown in Fig.1(c). If, however, the solute is of very high melting point, the M.P. effect exerts its influence, and the curves rise as in Fig.1(d). Of the transition elements, austenite stabilisers are in Groups lying to the right of iron in the Periodic Table. The present work has established this principle for the systems Fe-Ir[⊗], Fe-Pt[⊗], Fe-Au, Fe-Rh[⊗], Fe-Pd[⊗], Fe-Co, and Fe-Ni, and has shown that the systems Fe-Os[⊗] and Fe-Ru[⊗] also belong to this class - the symbol [⊗] means that the system was either incompletely known, or unknown when the present work was started. Fe-Mn is also an austenite stabiliser.

III.

On the experimental side, the present work has led to the accurate determination ($\pm 1^\circ\text{C}$) of the liquidus curves in the systems referred to below, and also to the determination of the solidus curves. The experimental errors for the latter are somewhat greater, and vary from one system to another. The work has been carried out by thermal analysis, except that the microscopical and quenching method had to be used in some systems for the determination of steeply falling solidus curves. The thermal analyses also led to the approximate determination of the A_4 transformation curves in the alloys.

Iron-Silver (W. Hume-Rothery and W.S. Gibson. For synopsis and reference to published paper, see Appendix A, No.1).

Iron and silver form immiscible liquids and no detectable solid solution. This may be connected with the fact that silver is almost exclusively univalent in its chemical compounds, with the result that electrons of a d-type are not available when it is alloyed with iron. This is in contrast with the behaviour of copper and gold which do dissolve to some extent in iron and exhibit higher valencies.

Iron-Palladium (W. Hume-Rothery and W.S. Gibson. For synopsis and reference to published paper, see Appendix A, No. 1).

The equilibrium diagram is of the type of Fig.1(c). Although still of favourable size factor, the atomic diameter of Pd is considerably larger than that of iron, and both δ - and γ - liquidus and solidus curves fall steeply.

Iron-Rhodium (W. Hume-Rothery and W.S. Gibson. For synopsis and reference to published paper, see Appendix A, No.1).

The equilibrium diagram is of the general form of Fig. 1(c) except that, whilst the δ -liquidus and solidus curves fall fairly steeply, the corresponding curves for the γ -phase rise slightly.

Iron-Ruthenium (W. Hume-Rothery and W.S. Gibson. For synopsis and reference to published paper, see Appendix A, No.1).

The high melting point of ruthenium results in the M.P. effect raising both δ - and γ -liquidus and solidus curves.

Iron-Technetium (W. Hume-Rothery and R.A. Buckley. For synopsis and reference to published paper, see Appendix A, No.2).

The effect of technetium is to produce a very slight rise in the δ -liquidus and solidus curves, and a slight fall in the A_4 points. The behaviour is intermediate between those of manganese and rhenium, and of molybdenum and ruthenium in the vertical and horizontal rows of the Periodic Table, respectively.

Iron-Molybdenum (W. Hume-Rothery, W.S. Gibson and J.R. Lee. For synopsis and reference to published paper, see Appendix A, No.3).

The first additions of molybdenum produce a fairly steep fall in the δ -liquidus and solidus curves, in agreement with the rather large size-factor. The curves then pass through a minimum which is at the same atomic % of molybdenum as that of chromium for the minimum in the system Fe-Cr. The present results suggest that there are low temperature transformations, and that the Fe-Mo diagram may be more complicated than is usually imagined.

Iron-Niobium (W. Hume-Rothery, J.R. Lee, and W.S. Gibson. For synopsis and reference to published paper, see Appendix A, No.3).

In the transition elements of the Second Long Period, niobium is on the borderline of the zone of favourable size-factor. In agreement with this, the δ -liquidus and solidus curves fall relatively steeply.

Iron-Zirconium (W. Hume-Rothery, W.S. Gibson, and J.R. Lee. unpublished).

(continued...)

Owing to the unfavourable size-factor of zirconium, the δ -liquidus and solidus curves fall steeply. The results so far obtained are shown in Fig.2. It is felt that some of these points are not entirely reliable, and it is hoped to do a little more work on this system before the results are published.

Iron-Gold (W. Hume-Rothery and R.A. Buckley. For synopsis and reference to published paper, see Appendix A, No.2).

The system Fe-Au resembles Fe-Cu, but the solid solutions in δ - and γ -iron are more restricted owing to the larger size-factor.

Iron-Platinum (W. Hume-Rothery and R.A. Buckley. For synopsis and reference to published paper, see Appendix A, No.4).

In spite of the rather large size-factor, the δ -liquidus and solidus only fall slightly, and the γ -liquidus and solidus are almost horizontal. This is the result of the M.P. effect. Platinum is a very strong γ -stabiliser.

Iron-Iridium (W. Hume-Rothery and R.A. Buckley. For synopsis and reference to published paper, see Appendix A, No.2).

Owing to the M.P. effect, both δ - and γ -liquidus and solidus curves are raised. Iridium is the strongest γ -stabiliser yet encountered.

Iron-Osmium (W. Hume-Rothery and R.A. Buckley. For synopsis and reference to published paper, see Appendix A, No.2).

Owing to the M.P. effect, both δ - and γ -liquidus and solidus curves rise very steeply.

Iron-Aluminium (J.R. Lee. For synopsis and reference to published paper, see Appendix A, No.5).

The liquidus and solidus curves for the complete system have been determined. The results show that there are two eutectics in the region 65 - 75 atomic % Al.

Iron-Gallium (W. Hume-Rothery and C. Dasarathy. Work still in progress.)

For the iron-rich half of the diagram, the results are very much like those of the system Fe-Al. There are, however, complications in the α - δ region, and a face-centred cubic phase has been found. The present state of this work is shown in Fig.3. IT IS PARTICULARLY REQUESTED THAT THIS DIAGRAM IS TREATED AS CONFIDENTIAL UNTIL THE WORK IS FINISHED.

Iron-Carbon (W. Hume-Rothery and R.A. Buckley. For synopsis and reference to published paper, see Appendix A, No.6).

This work has established the diagram conclusively, and shown that Hansen's selection from the conflicting data was essentially correct.

Iron-Carbon-Nickel (W. Hume-Rothery and R.A. Buckley. Work still in progress).

The liquidus and solidus points have been determined for a wide range of compositions, and the results obtained are shown in Fig.4. Attempts are now being made to determine the tie lines connecting the compositions of liquid and solid phases in equilibrium at 1450°C. For this purpose specimens are being quenched from the 2-phase region, and the compositions of the chilled liquid and of the solid phase are being determined by electron-probe methods. The first results were unsatisfactory, but better preparation of the surfaces should improve things, and a number of consistent tie-lines has been obtained. This work is being continued, and we have to thank the British Iron and Steel Research Association, and the Associated Electrical Industries Limited for allowing access to their apparatus. IT IS REQUESTED THAT FIGURE 4 BE TREATED AS CONFIDENTIAL UNTIL THE WORK IS COMPLETE.

High Purity Iron

By the kindness of the Battelle Memorial Institute and Mr. James W. Halley of the Inland Steel Company, Indiana

Harbor Works, East Chicago, Indiana, we were supplied with some iron of very high purity. Experiment showed that the freezing point of this material was indistinguishable from that of the 99.96 - 99.97% metal supplied by the British Iron and Steel Research Association and it was, therefore, thought unjustifiable to use more of the very pure metal for the constitutional work. The metal was given to my colleague Dr. J.W. Christian and is being used in his research work on tension and compression tests to measure the yield point and flow stress at various strain rates and temperatures from 2°K. to 300°K.

IV.

The work referred to in Section III enables the $\delta/(\delta + \gamma)$ and $(\delta + \gamma)/\gamma$ solid solubility curves to be drawn in the systems concerned. From the gradients of these at zero atomic % of solute, it is possible to calculate the free-energy of transfer, ΔG , of 1 mole of the solute element from the γ - to the δ -phase on the assumption that the curves follow the laws of dilute solutions. Fig.5 shows the ΔG values for the $\delta \rightleftharpoons \gamma$ transformation at the A_4 points; with the convention that a +^{ve} sign indicates a ferrite stabiliser. In this way a scale is obtained of the relative effects of the different solutes in stabilising the δ - or γ -phases, and the following points may be noted:

- (a) The effect of Mn in the First Long Period is anomalous, but for the remaining elements the new data at the A_4 point confirms the general principle that transition elements to the left of iron in the Periodic Table are ferrite stabilisers, whilst those to the right are austenite stabilisers. On the other hand, at the A_3 point exceptions are shown by Cr, Mn, and Co, since the A_3 point

is lowered by Mn and Cr, and raised by Co.

- (b) The greatest austenite stabilisation is shown by Os-Ir-Pt, and in Group VIII the element showing the greatest austenite stabilisation moves from Group VIIIC (Ni) $\rightarrow$ VIIIB (Rh) $\rightarrow$ VIIIA (Os) on passing from the First to the Second to the Third Long Periods.

These effects have been discussed in the published paper by Hume-Rothery and Buckley of which the synopsis and reference are given in Appendix A, No.7.

V.

Just as the gradients of the $\delta/(\delta + \gamma)$ and $(\delta + \gamma)/\gamma$ phase boundaries may be analysed so as to give the ΔG values referred to above, so the gradients of the liquidus and solidus curves may be used to calculate the free energy of transfer (ΔG_Y) of 1 mole of solute element from the solid to the liquid solution. In systems where the liquidus and solidus are raised, ΔG_Y is negative, whilst positive values of ΔG_Y mean that the solute lowers the liquidus and solidus. The ΔG_Y values obtained in this way are shown in Fig.6. In the First Long Period, the effect of Mn is abnormal, but otherwise there is a clear general tendency to show a minimum value of ΔG_Y in Group VIIA. For the elements of Group VIII in any one Period, the effects are remarkably regular, and the points for Fe-Co-Ni, Ru-Rh-Pd, and Os-Ir-Pt are on almost straight lines. For the elements of the earlier Groups, the high positive values of ΔG_Y for Ti, Zr, Ta, and Nb are clearly the result of the large size-factors.

The points in Fig.6 place the elements in the order of their liquid stabilising powers relative to δ -iron, in the sense that a high positive value of ΔG_Y favours the liquid state. The values clearly involve both lattice distortion, Group Number or valency effects, and the M.P. effect. Since

the various elements crystallise in different structures, it is not possible to determine the M.P. effect accurately, but Hume-Rothery and Buckley (Appendix A, No.6) have shown how an approximate estimation of the M.P. effect may be made, and in this way it is possible to estimate the part of ΔG_Y which is due to lattice disturbing effects (size-factor, Group Number Difference, etc.). If we use the symbol ΔF_Y for the contribution of lattice disturbing effects to ΔG_Y , Fig.7 shows the values of ΔF_Y for the different elements. It will be seen that Mn now lies in sequence, and that the values of ΔF_Y for the elements of the First Long Period are systematically less than those of the Later Periods, as would be expected from the size-factors and electronic factors. For solutes from the Second and Third Long Periods, the point for Zr appears anomalous, and it is not possible to say whether this is a genuine effect, or is due to an error in the determination of a steeply falling solidus. These data have been discussed by Hume-Rothery and Buckley (Appendix A, No.6).

Fig.8 shows the equilibrium diagrams of the alloys of iron with the transition elements, and it is the slopes of the liquidus and solidus curves on the one hand, and of the A_4 transformation curves on the other, which are generalised by the values of ΔG , ΔG_Y , and ΔF in Figs.5, 6, and 7. In this way the research project has succeeded in revealing some of the principles underlying the equilibrium diagrams of this series of alloys.

VI.

The liquidus and solidus curves of some of the above systems have been examined thermodynamically. It has been shown that for the system Fe-Mn, both δ and γ liquidus and solidus curves obey the laws of dilute solutions, and for

the γ -phase these hold up to surprisingly high concentrations. In the system Fe-Ni there is again good agreement, and the data so far examined suggest that all the alloys are monatomic in both solid and liquid solutions. In the system Fe-C, the γ -liquidus and solidus do not agree with the laws of monatomic dilute solutions, and the data lend support to the view that, in the liquid phase, carbon is present in the form of Fe_3C molecules. This work is being continued.

VII.

During the period of the contract, W. Hume-Rothery has been writing a book on the Structure of the Alloys of Iron, some of which will deal with the research work of the present contract. He has also lectured at Sheffield and at Dounreay on the Structure of Iron Alloys, and has attended conferences on the Electron Theory of Metals (Physical Society 1958), Magnetism (Oxford 1959), and on Constitutional Research (Institute of Metals 1961). By the kindness of the United States Air Force, W. Hume-Rothery and J.W. Martin visited the U.S.A. and Canada in 1960 and lectures were given and discussions held at the National Research Council, and the Department of Mines and Technical Surveys, Ottawa; the Climax Molybdenum Company, Detroit; the Wright Air Development Center, Dayton; the Carnegie Institute of Technology, and the Mellon Institute, Pittsburgh; Oak Ridge National Laboratory, Oak Ridge; the National Bureau of Standards, and the Naval Research Laboratory, Washington D.C. This trip was of great interest and value to both visitors. W. Hume-Rothery also lectured on Transition Metals to the A.I.M.E. in New York (1962).

The present research work has been made possible by grants from both the U.S.A.F. and the British Iron and Steel Research Association, and all those concerned must express their thanks for the help received.

APPENDIX A

1. The Constitution of Alloys of Iron with Ruthenium, Rhodium, Palladium and Silver.

J. Iron and Steel Institute, Vol.189, p.243, July 1958.
W.S. Gibson and W. Hume-Rothery.

Iron-rich binary alloys with ruthenium, rhodium, palladium, and silver have been investigated by thermal analysis in the temperature range 1380 - 1560°C.

The equilibrium diagrams of the systems Fe-Ru, Fe-Rh, and Fe-Pd are of the expanded γ -field type, in which the temperatures of the A_4 transformations are raised by the addition of solute. In the system Fe-Ru, the body-centred cubic (δ) iron-rich solid solution is formed by a peritectic reaction between the liquid and the face-centred cubic (γ) iron-rich solid solution. In the systems Fe-Rh and Fe-Pd, the face-centred cubic (γ) solid solution is formed peritectically from liquid and δ -solid solution. No appreciable solid solution of silver in iron at high temperatures was detected.

2. Liquidus $\rightleftharpoons$ Solidus Relations in Iron-rich Alloys of the Systems Fe-Tc, Fe-Os, Fe-Ir, and Fe-Au.

J. Iron and Steel Institute. To be published in 1963.
R.A. Buckley and W. Hume-Rothery.

The liquidus, solidus, and A_4 transformation temperatures have been determined for iron-rich alloys of the systems Fe-Tc, Fe-Os, Fe-Ir, and Fe-Au. In the system Fe-Tc the δ -liquidus curve is very nearly horizontal, and Tc acts as a very weak ferrite-stabiliser. In the systems Fe-Os, and Fe-Ir, both solutes act as austenite stabilisers, and the δ - and γ -liquidus and solidus curves are raised by the addition of solute. The iron-rich part of the equilibrium diagram of the system Fe-Au resembles that of Fe-Cu, except that the extent of the solid solution of Au in γ -iron is smaller, as would be expected from the atomic size factors.

3. Liquidus-solidus relations in iron-rich iron-niobium and iron-molybdenum alloys.

J. Iron and Steel Institute, Vol. 198, p. 64, May 1961.
W.S. Gibson, J.R. Lee and W. Hume-Rothery.

The liquidus and solidus relations in iron-rich alloys of the systems iron-niobium and iron-molybdenum have been determined for alloys of high purity. Some information has also been gained of the constitution of the alloys in the solid state. For the system Fe-Nb, the general form of the earlier diagram is confirmed, but the composition of the δ -eutectoid is at a lower percentage of niobium than was previously imagined. For the system Fe-Mo, the general form of the earlier diagram is again confirmed, but the freezing range of the δ -phase alloys is narrower than was previously supposed. In the systems Fe-Cr and Fe-Mo the δ -liquidus and solidus curves pass through minima at almost the same composition (c. 20 at.-% solute) in spite of the larger size of the molybdenum atom.

4. Liquidus and solidus relations in iron-rich iron-platinum alloys.

J. Iron and Steel Institute, Vol. 193, p. 61, Sept. 1959.
R.A. Buckley and W. Hume-Rothery.

The liquidus and solidus curves of Fe-rich Fe-Pt alloys: have been determined accurately by thermal analysis, and approximate values obtained for the δ/γ transformation lines. The system is of the open γ -field type, and is remarkable for the extremely narrow freezing range of both δ - and γ -solid solution alloys. There is a $(\delta + \text{Liq}) \rightleftharpoons \gamma$ peritectic horizontal at 1519°C, and with increasing Pt-content the γ -liquidus and solidus probably fall to a very shallow minimum. Comparison of the equilibrium diagrams of the systems Fe-Pt, Fe-Pd, and Fe-Ni shows that Pt has a remarkably high effect in stabilizing the γ -phase.

5. Liquidus-solidus relations in the system iron-aluminium.

J.Iron and Steel Institute, Vol.194, p.222, Feb.1960.
J.R. Lee.

The liquidus and solidus curves for the system iron-aluminium have been determined accurately by thermal analysis. The addition of aluminium to iron produces a very slight rise in the freezing point but it is not possible to say whether this is a characteristic of the true binary equilibrium diagram or whether it is due to the removal of the last traces of oxygen from the high-purity iron which was used for the experiments. The system contains two eutectics in the region 65 - 75 at.-% Al, and the previously accepted diagram is shown to be incorrect in indicating a peritectic horizontal.

6. Liquidus and solidus relations in iron-rich iron-carbon alloys.

J.Iron and Steel Institute, Vol.196, p.403, Dec. 1960.
R.A. Buckley and W. Hume-Rothery.

The equilibrium diagram of iron-rich iron-carbon alloys has been determined by a combination of the methods of thermal analysis and microscopical examination of quenched specimens. The results are in good agreement with the curves given in other recent work, and confirm that Hansen was correct in rejecting experimental points which made the equilibrium diagram appear to be so uncertain.

7. The liquid $\rightleftharpoons$ solid and $\delta \rightleftharpoons \gamma$ equilibria in iron-rich alloys.

J. Iron and Steel Institute. To be published in 1963.
R.A. Buckley and W. Hume-Rothery.

The relative effects of various alloying elements on the liquid $\rightleftharpoons$ solid and δ/γ transition points in iron alloys are described and classified. The importance of melting point and size factor differences between solute and solvent elements in determining the effects on the liquid $\rightleftharpoons$ solid equilibrium is verified. The liquid or solid phase stabilising power of a solute element in iron is assessed in terms of the free energy difference (ΔG_Y) of the elements between the two phases. A systematic variation of ΔG_Y with Group Number in the Periodic Classification is found. The proportion of ΔG_Y due to melting point differences is assessed and the significance of the remaining energy difference (ΔF_Y) is discussed.

A similar treatment is applied to the $\delta \rightleftharpoons \gamma$ phase transformation. It appears that electronic factors are more important than size factors in this case in determining which structure is stable. A possible explanation is presented in terms of the degree of s and d hybridisation of the bonding electrons in the solute atoms.

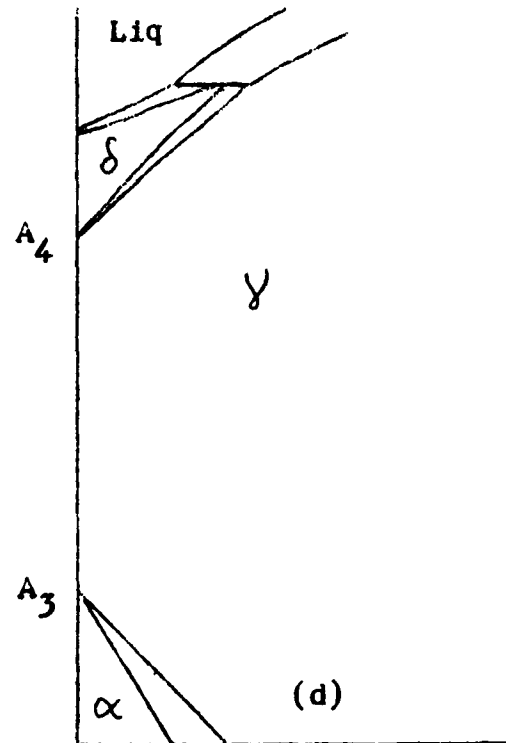
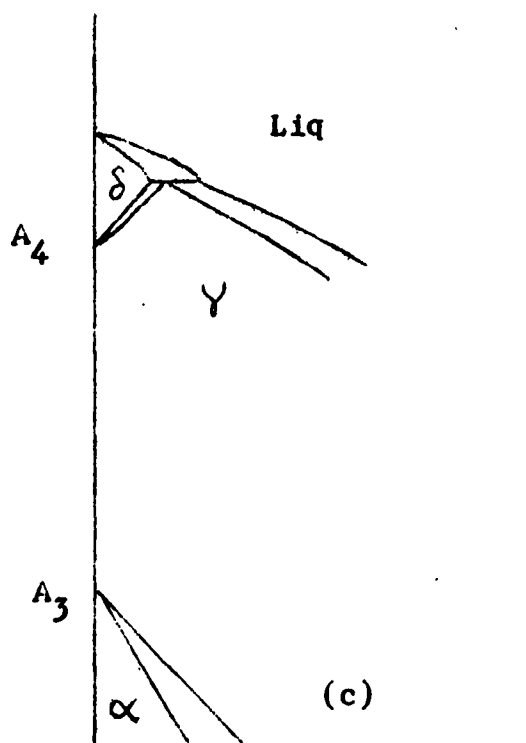
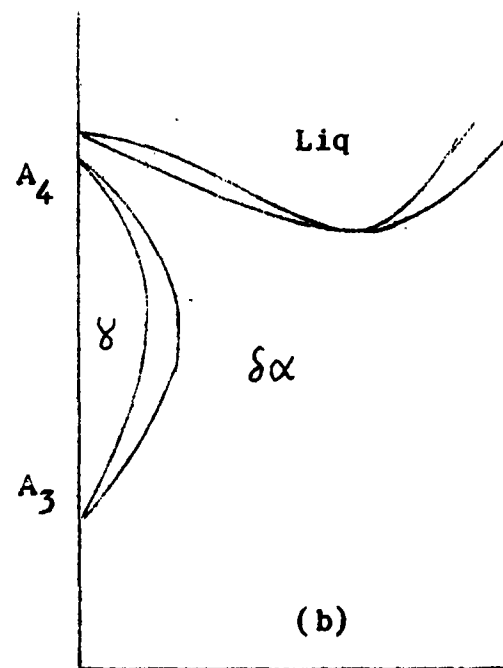
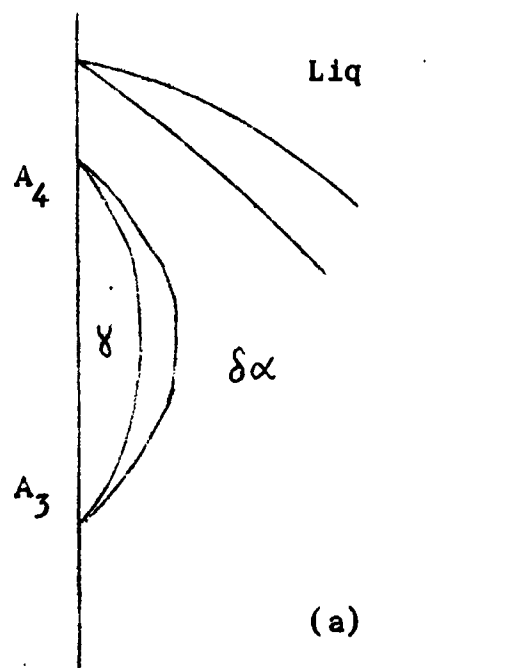


Fig. 1.

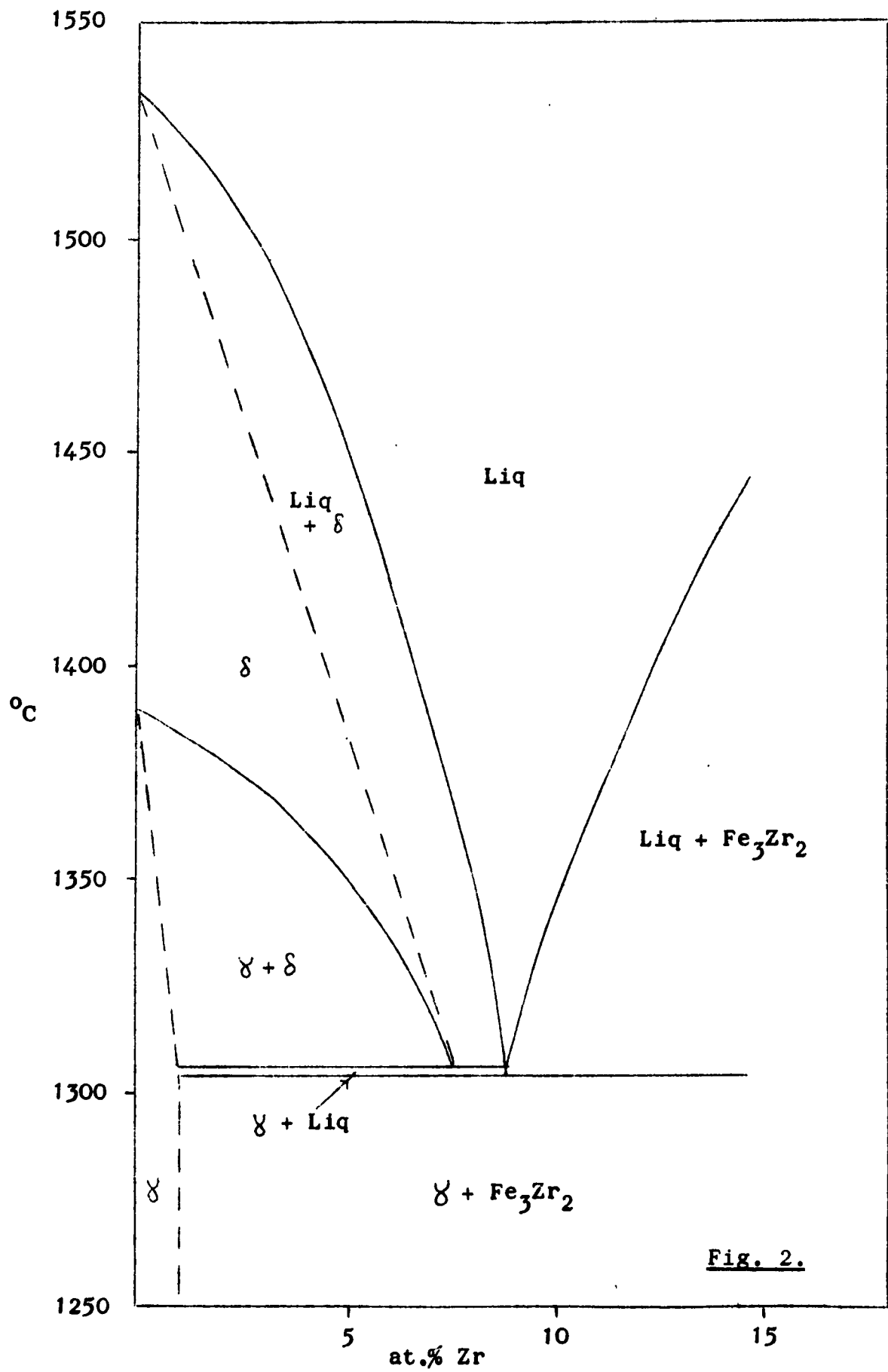


Fig. 2.

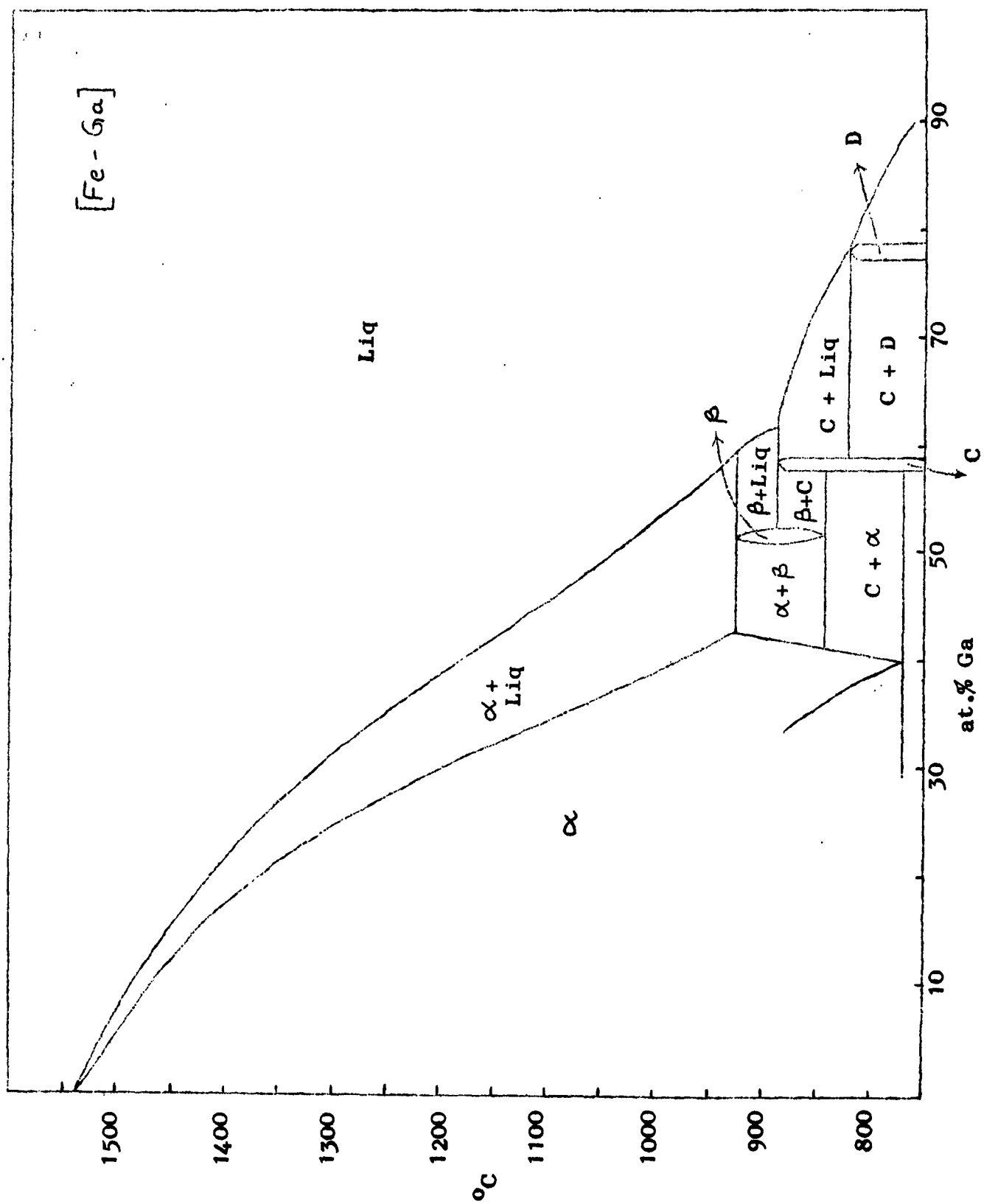


Fig. 3.

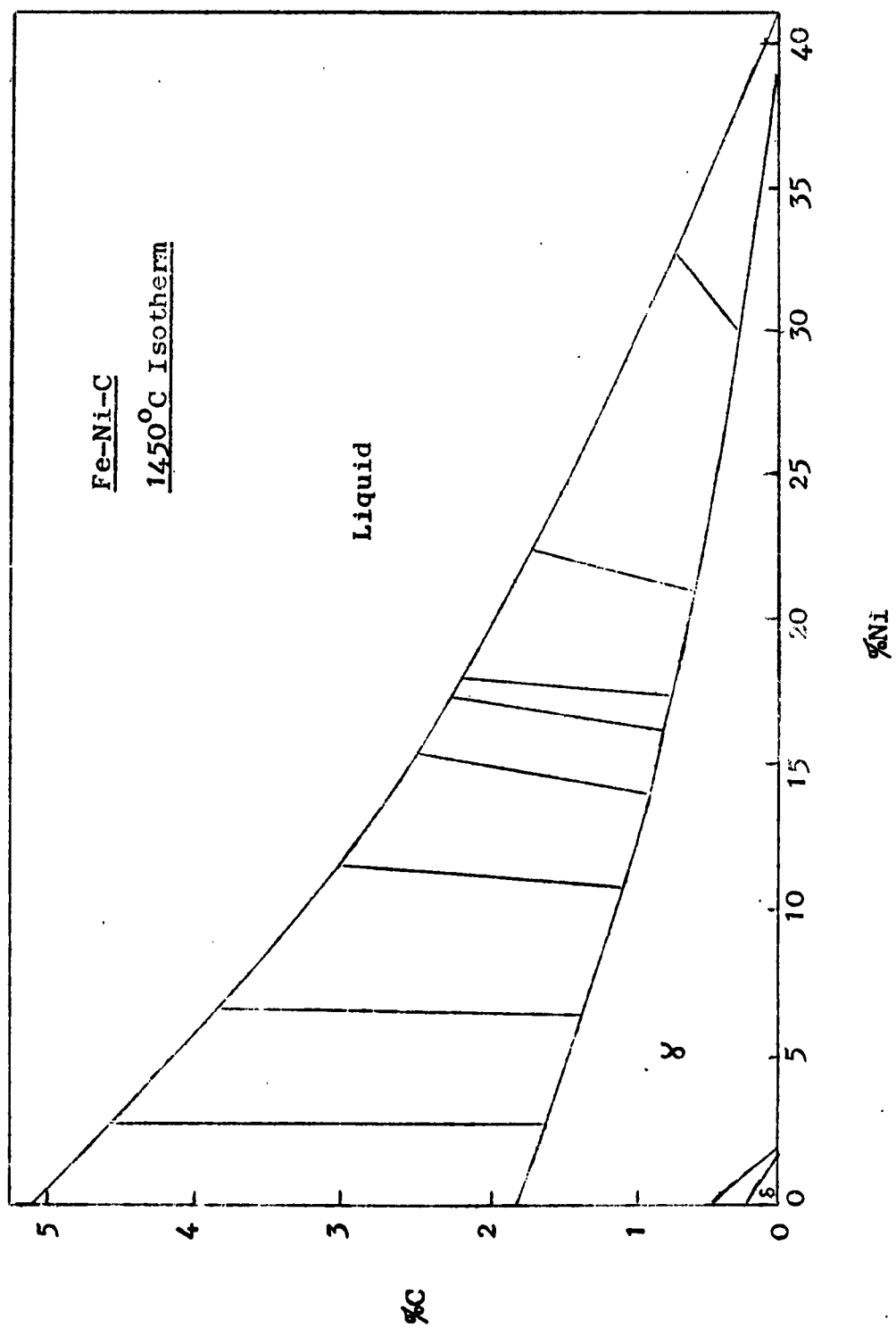


Fig. 4. (a)

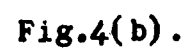


Fig.4(b) .

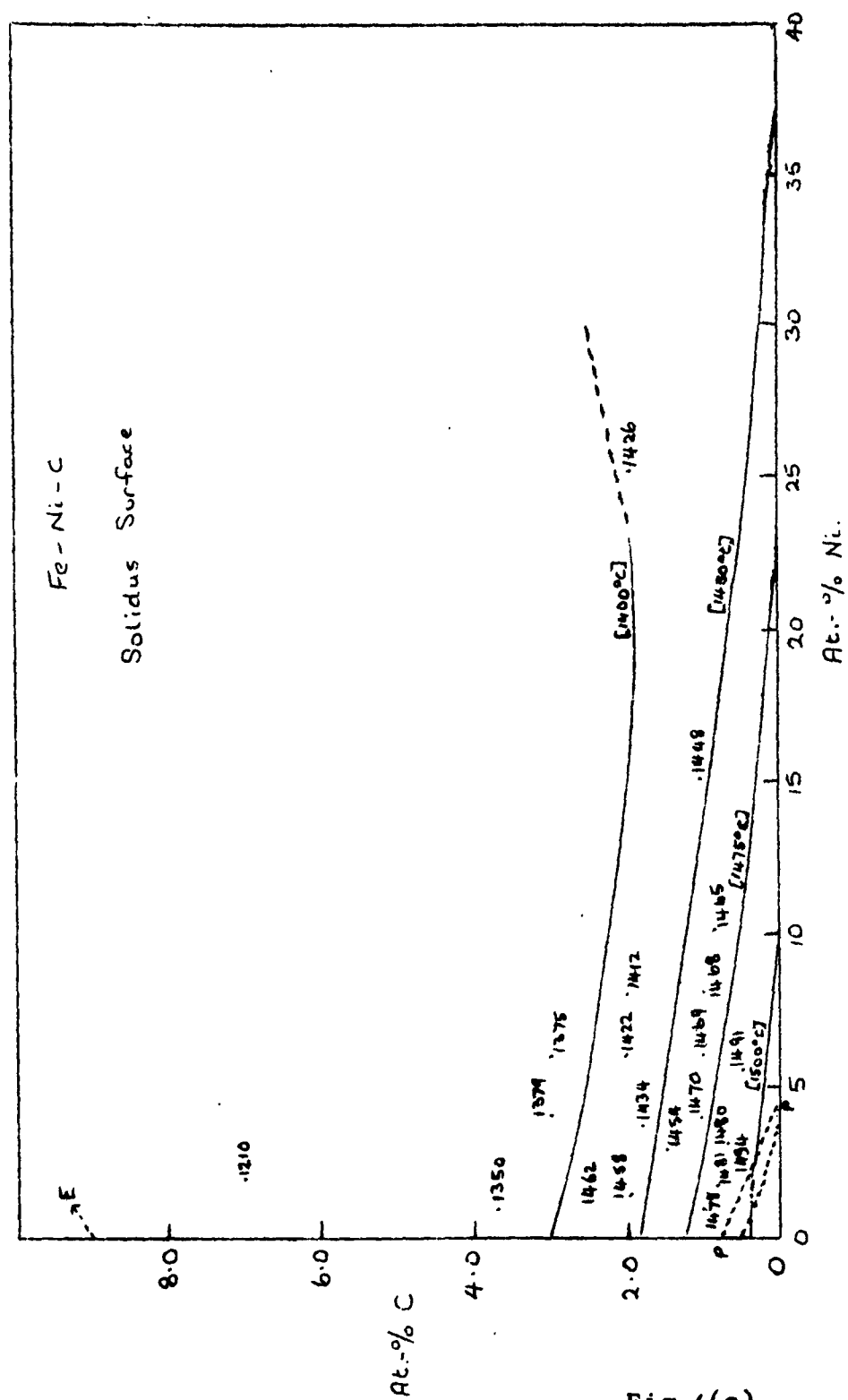


Fig.4(c).

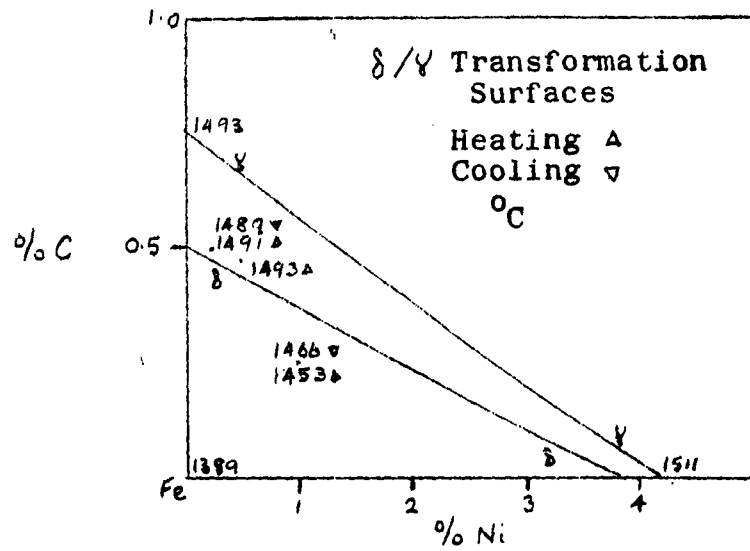


Fig.4(d).

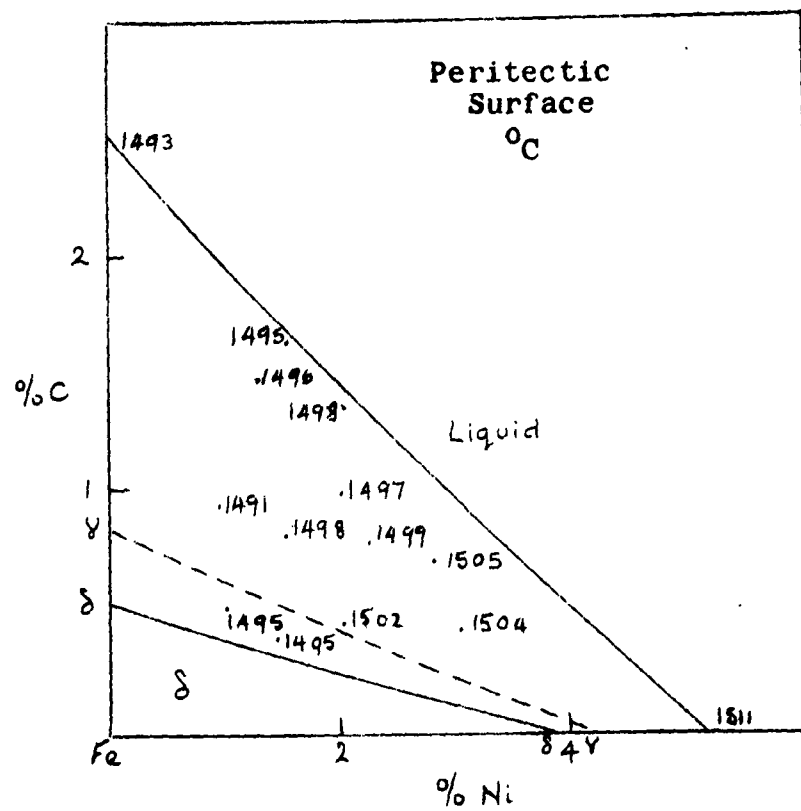


Fig.4(e).

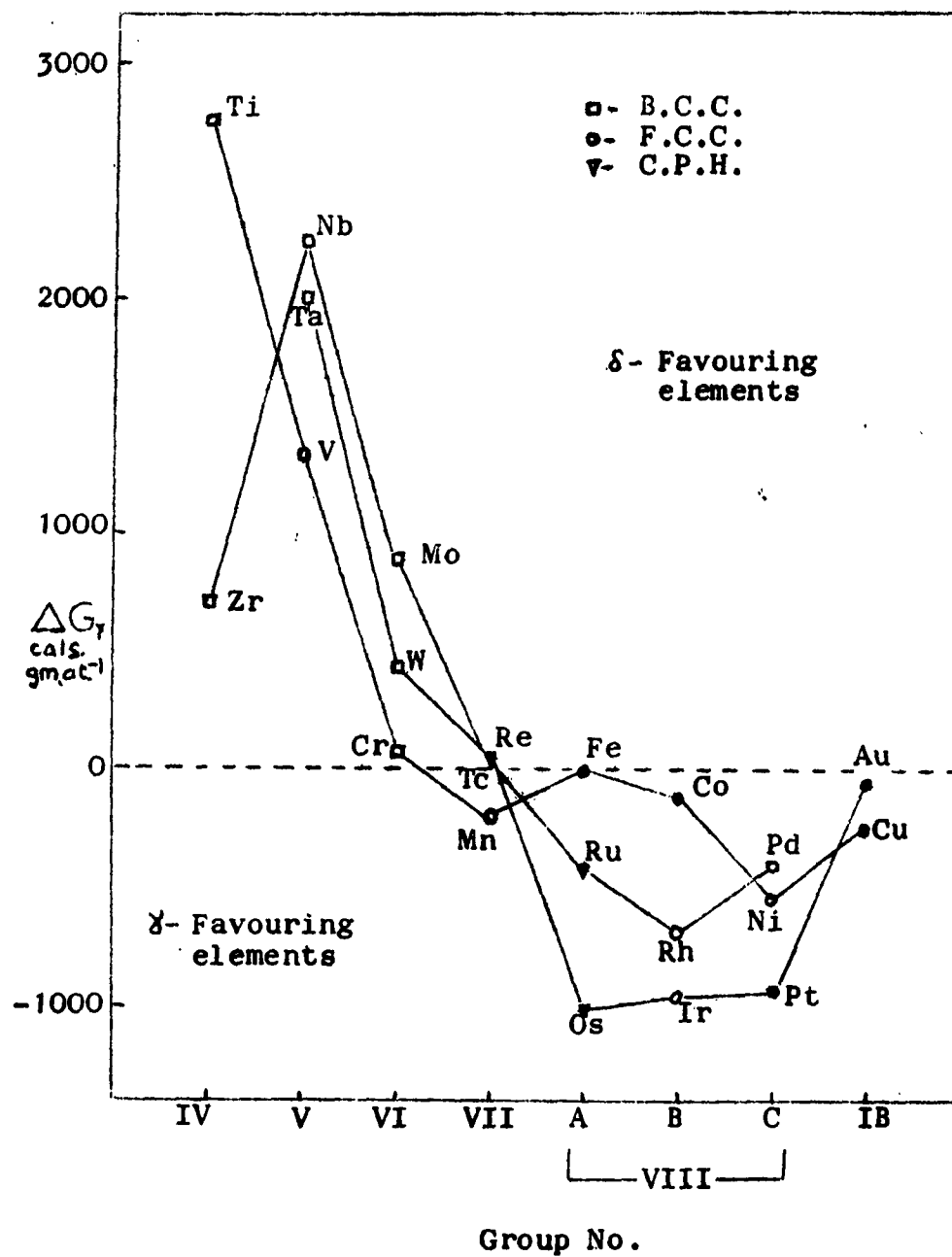


Fig. 5.

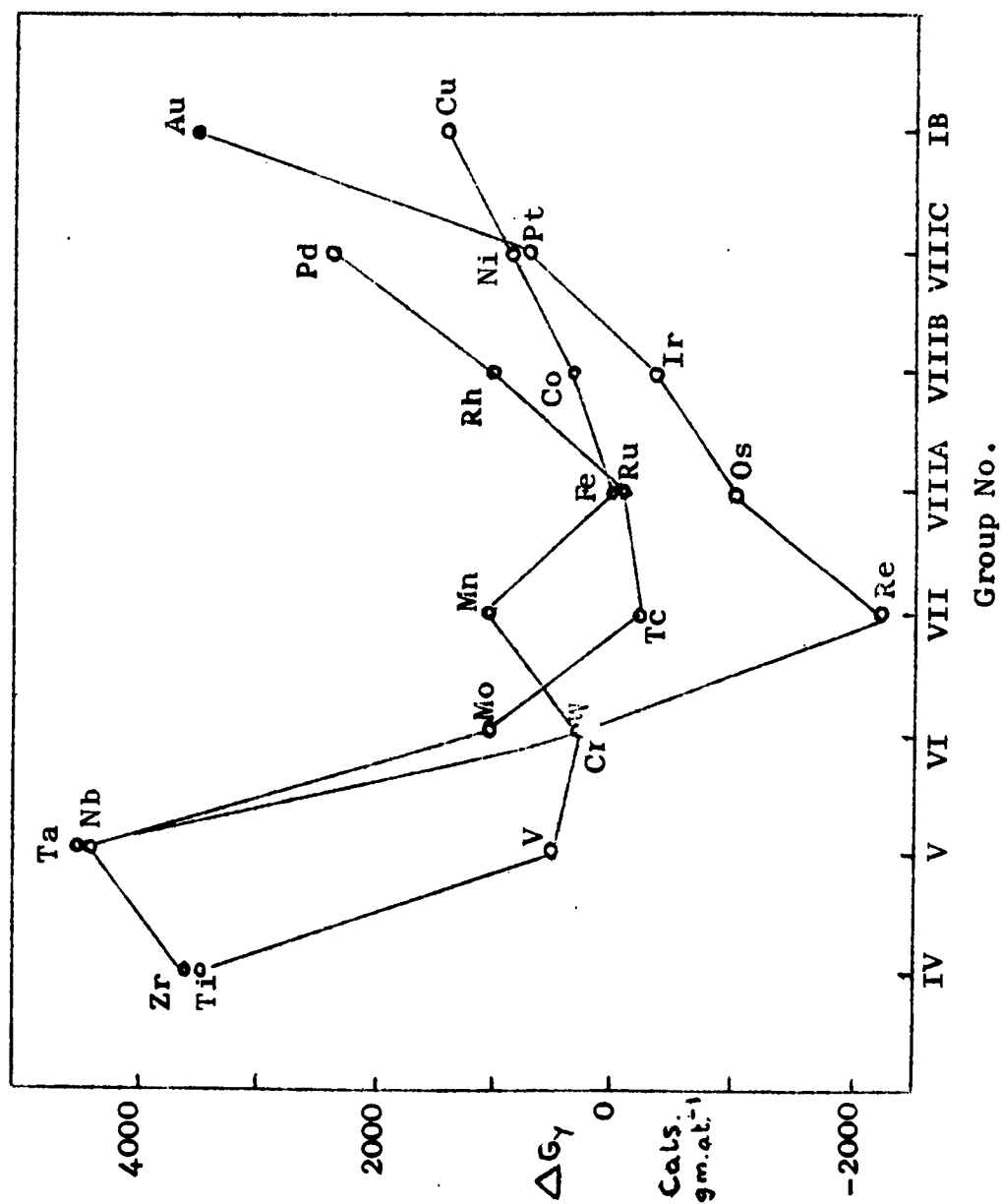


Fig. 6.

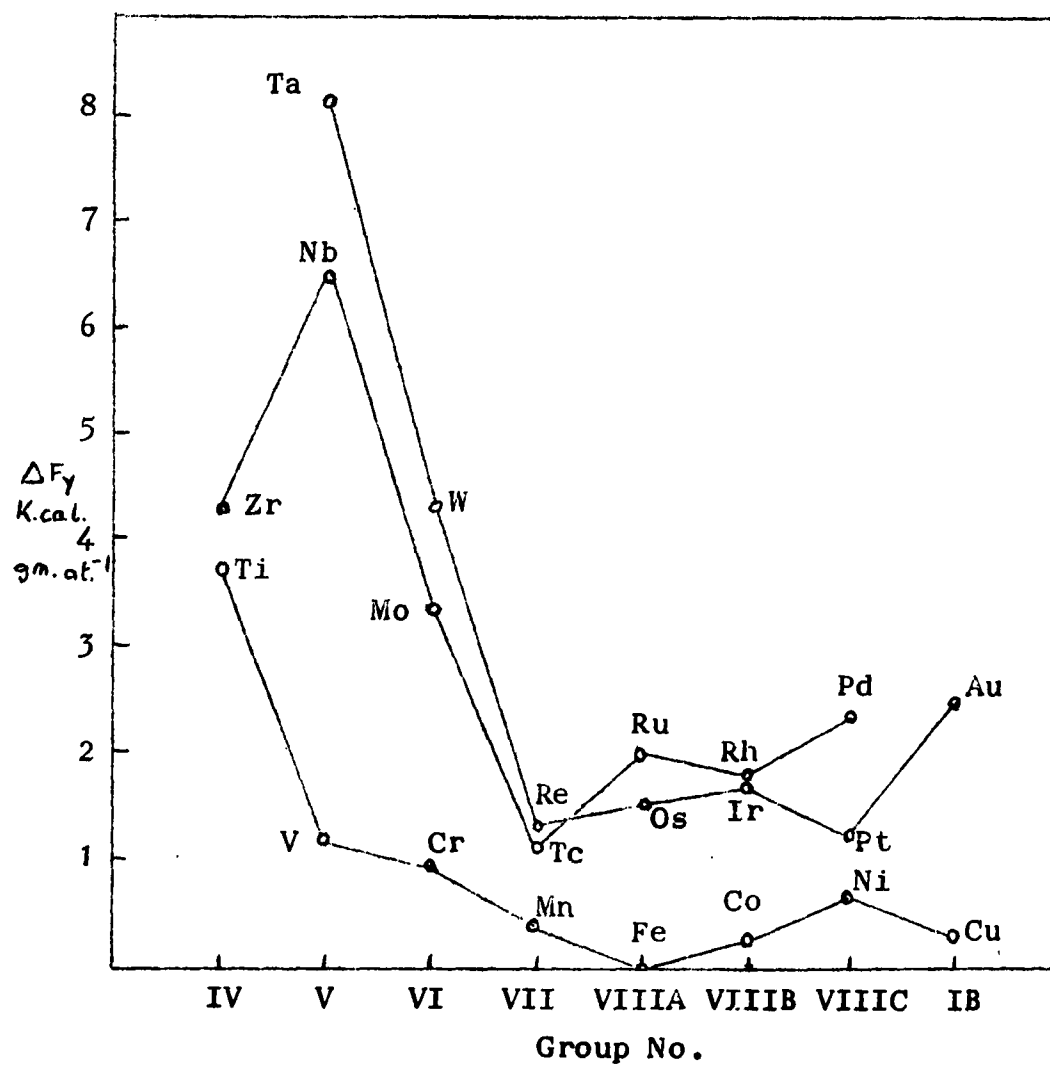


Fig. 7.

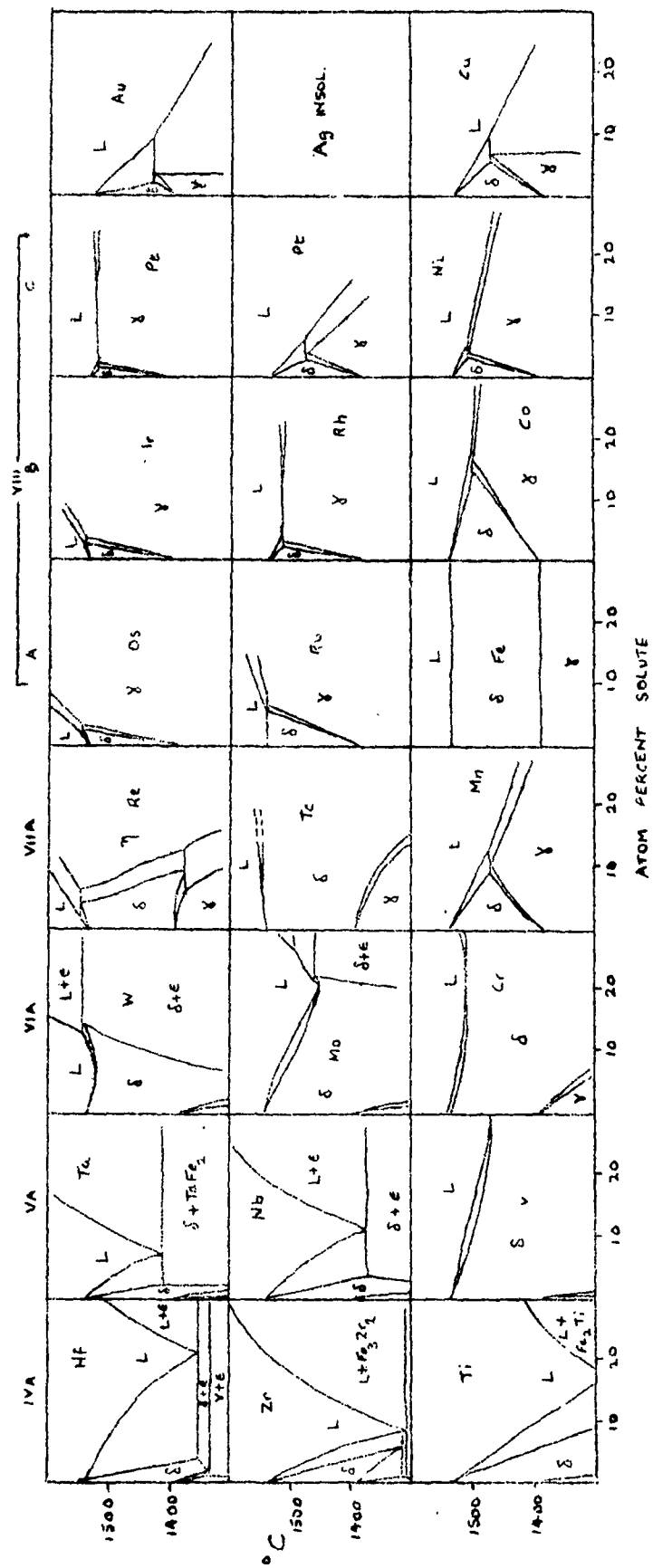


Fig. 8.

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United States Air Force, Office of
Aerospace Research, European Office,
Brussels, Belgium.

Technical Report Nr. Final.

RESEARCH ON THE FREEZING AND MELTING
POINTS OF IRON ALLOYS. W. Hume-Rothery
Date: November 1962

Nr. pages 11, and Illus.8, and Appendix.

Organization: University of Oxford,
England. (Department of Metallurgy)

ABSTRACT: The liquidus, solidus and A_4
transformation points have been deter-
mined for binary iron-rich alloys with
C, Al, Ga, Zr, Nb, Mo, Tc, Ru, Rh, Pd,
Ag, Os, Ir, Pt, and Au. In the systems
Fe-Al and Fe-Ga, liquidus and solidus

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United States Air Force, Office of
Aerospace Research, European Office,
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Technical Report Nr. Final.

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