Ferromagnetic GoldIron Alloys

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Ferromagnetic Gold-Iron Alloys

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Magnetic measurements were made on a gold-iron alloy containing 37 atomic percent iron, both in the quenched and successively annealed states. It was found that the quenched supersaturated solid solution is ferromagnetic. When this supersaturated solid solution was gradually heated up, perceptible precipitation took place at about 450°C. Both the final gold-rich and iron-rich phases precipitated at 450°C were found to be magnetic at room temperatures. Magnetic measurements, checked by Debye-Scherrer patterns, indicate that the mechanism of precipitation in this alloy is mainly of the non-uniform type.

SEVERAL investigations on the gold-iron alloys are available in the literature. These results have been summarized by Hansen[1] in a phase diagram which is reproduced in Fig. 1. While this diagram does not show whether the gold-rich alloys are magnetic, Shih[2] and Jette and collaborators[3] found that gold-rich alloys containing 10 and 15 percent iron are definitely ferromagnetic at room temperatures. In the course of experiments on the aging of supersaturated solid solutions, we have had occasion to prepare an alloy of gold containing 37 atomic percent iron. Our main purpose was to investigate the mechanism of precipitation in this alloy and the change of magnetic properties with composition and other effects.

The alloy was made by melting pure gold and iron, covered by borax, in a porcelain crucible in a gas furnace and casting in an iron mold in the form of a $\frac{7}{16}$-inch rod. The ingot was then swagged into a $\frac{1}{4}$-inch rod. All the specimens subsequently used were cut from this quarter-inch rod and cold worked and heat treated. Chemical analysis gave the accurate composition as 13.74 weight percent iron.

The apparatus and the method of high temperature measurements were the same as those described in a previous work on copper-iron alloys reported by two of the authors.[4] With this apparatus, we were able to separate the paramagnetic susceptibility superposed on the ferro-magnetic saturation magnetization. As the alloy was magnetic in this case, the force exerted by the field on a sample was so large that very small specimens had to be used. In each case, the specimen was about 1 mm in diameter and 3 mm long. The specimen was placed in a copper holder which was suspended from the balance arm. The force exerted by the field on the holder and suspensions was found to be negligible.

For liquid nitrogen measurements, the tube furnace was replaced by a long Dewar flask of about the same size. Inside the Dewar flask was a long Pyrex tube which was closed at the bottom and in which the specimen was suspended. Liquid nitrogen was introduced into the
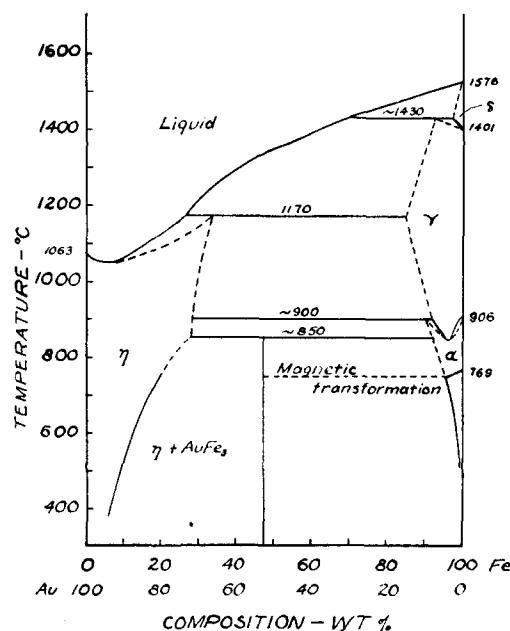
[1] M. Hansen, *Der Aufbau der Zweistofflegierungen* (Springer, Berlin, 1936).

[2] J. W. Shih, Phys. Rev. **38**, 2051 (1931).

[3] E. R. Jette, W. L. Bruner, and F. Foote, Trans. A. I. M. E. **111**, 354 (1394).

[4] F. Bitter and A. R. Kaufmann, Phys. Rev. **56**, 1044 (1939).

FIG. 1. The gold-iron phase diagram according to Hansen.

space between the tube and the Dewar flask. Through a side opening at the top of the Pyrex tube, hydrogen could be blown into the latter in order to prevent condensation of air or water vapor inside the tube. The temperature was measured by a copper-constantan thermocouple made of fine wires, which was soldered to the specimen holder. Thus, the couple wires served as suspension for the holder at the same time. Low temperature measurements were also made by previously cooling the Dewar flask and the specimen with liquid nitrogen and measuring in steps as the temperature gradually rose. The drifting of the temperature was sufficiently slow so that during each set of measurements it was constant within a few degrees. At low temperatures, the force arising from the saturation magnetization became so large that the sensitivity of the balance had to be reduced. Hence, these measurements are not accurate enough for calculating the superposed paramagnetic susceptibilities.

By a simple arrangement, specimens could be annealed in hydrogen and quenched into water without being exposed to air. A piece of the sample thus quenched from 870°C was found to be still magnetic. Polishing and etching by aqua regia revealed under the microscope a polygonal structure with twins characteristic of a single phase. No trace of precipitate could be detected, thus proving the magnetic nature of the supersaturated solid solution.

Preliminary measurements on a quenched specimen showed that the supersaturated solid solution had a Curie temperature of about 230°C. Pronounced precipitation took place at 450°C on heating and at 500°C on cooling. The progress of precipitation could be watched by noticing the increase in the force exerted by the field on the specimen with time. In the temperature range 650°C–950°C in which the solid solution was paramagnetic, the susceptibility was found to obey the Curie-Weiss law. When precipitation was allowed to be fairly completed at 500°C, it was found that the specimen contained two phases both of which were ferromagnetic. The main phase had a Curie temperature near room temperature, while the precipitated phase had a Curie temperature above the equilibrium temperature of the solid solution.
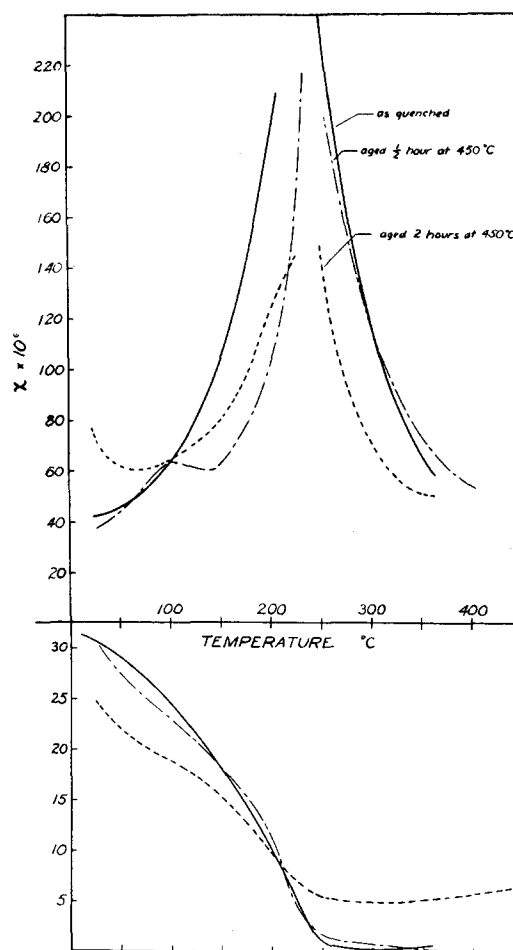

FIG. 2. Magnetic measurements on quenched and aged specimens of same alloy.

The results on three specimens with different treatments are shown in Fig. 2 in which Σ and χ are the mass saturation magnetization and the mass susceptibility, respectively. It is evident that all three specimens contain a phase with a Curie temperature of 230°C, which is better determined by the peak of the $\chi - T$ curve. This phase is easily identified as the undecomposed supersaturated solid solution. However, in the curves of the aged specimens, two additional phases show up, one of which is identified by the peaks of the $\chi - T$ curves near room temperature. The other phase, owing to its high Curie temperature and its small quantity, can only be detected in the $\Sigma - T$ curve of the specimen annealed for 2 hours at 450°C. These phases are evidently the products of precipitation. The
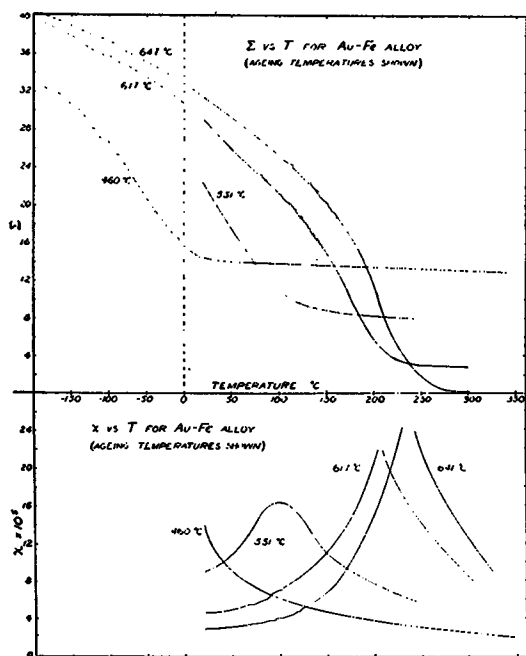
FIG. 3. Magnetic measurements on specimens which are approximately in equilibrium conditions at indicated temperatures.

presence of the precipitated phases with the original supersaturated solid solution in the same specimen characterizes the non-uniform nature of the precipitation process. In other words, precipitation in this alloy does not start at every point of the specimen at the same time and when precipitation occurs at any point the supersaturated solution breaks into the final phases with little intermediate change of composition.

Figure 3 shows the results on four specimens each annealed at a particular temperature for four days. These curves, however, should be interpreted differently. As the time of annealing was very long, equilibrium must have been approached in each specimen. Therefore, these curves show the variation of the Curie temperature of the gold-rich phase with composition rather than the mechanism of precipitation. The specimen annealed at 641°C still has a Curie temperature of 230°C, hence 641°C must be very near to the solubility limit temperature of the solid solution. One can approximately determine the variation of the Curie temperature of the gold-rich phase with composition by combining our results with Jette's[3] solubility limit curve. It is thus estimated that the Curie temperature becomes 0°C when the iron content is 8 percent by weight.

A portion of the sample was further swagged and drawn into a 0.006-inch wire. A piece of this wire was annealed and quenched from 970°C. The other was slowly cooled from 950°C with a duration of four hours in the range 500–400°C. Figures 4 and 5 show Debye-Scherrer patterns for the quenched and annealed wires, respectively. The quenched specimen gives spotted rings corresponding to an f.c.c. structure of lattice constant 3.967A. The annealed specimen, however, gives two sets of rings, one spotted and one fine, both corresponding to f.c.c. structures. The spotted set gives the same lattice constant as that of the quenched specimen while the fine rings give a lattice constant of 4.043A.
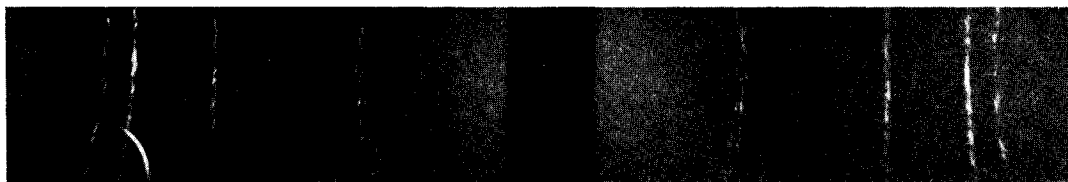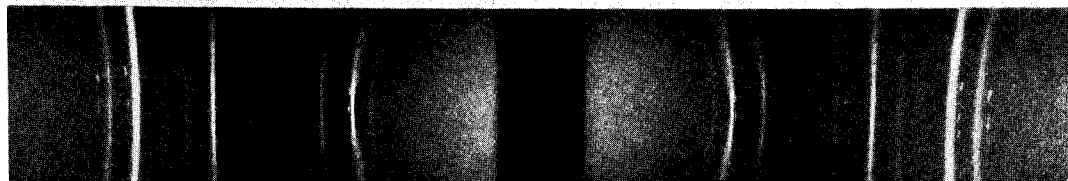

FIG. 4.

FIG. 5.

FIG. 4. Debye-Scherrer pattern of a specimen quenched from 970°C, showing a single phase of lattice constant 3.967A.

FIG. 5. Debye-Scherrer pattern of a specimen slowly cooled from 970°C, showing the original coarse-grained phase and a fine precipitated phase of lattice constant 4.043A.

These values are believed to be accurate to about 0.001A. According to Jette[3] and co-workers, the lattice constant of 4.043A corresponds to a gold-iron solid solution containing 3 percent of iron by weight. This seems to disagree with our results hitherto described, as we always found in an annealed specimen a phase with a Curie temperature near room temperature which, presumably, contained more than 8 percent of iron. The explanation is that, during cooling, the annealed specimen in this case was left at 340°C overnight so that the precipitated gold-rich phase should almost be in equilibrium at that temperature. If we check up with Jette's[3] solubility limit curve again, we find that the solubility of iron in gold at 340°C is just about 3 percent. The annealed specimen for x-ray investigation was later checked by magnetic measurements. The results are about the same as shown by the curves in Fig. 2. The only difference is that, in this case, we no longer find any Curie point near room temperature.

Thus, both magnetic and x-ray measurements prove the non-uniform nature of the precipitation process in the gold-iron alloys. The absence of the precipitated iron-rich phase in the Debye-Scherrer pattern of the annealed specimen shows that its quantity must be very small. This supports the conclusion obtained by Jette[3] and co-workers that the intermetallic compound AuFe$_3$ in Hansen's[1] phase diagram does not exist and that the solubility of gold in α-iron is very small. The fact that the lines of the precipitated gold-rich phase are fine in contrast with the spotted lines of the original supersaturated phase also substantiates the conclusion arrived at by previous investigators[5] that in non-uniform precipitation, the original grains in the sample are not preserved.

In conclusion, the authors wish to express their appreciation to Dr. B. M. Loring for taking the x-ray pictures.

[5] R. Becker and W. Döring, *Ferromagnetismus*, Sections 26 and 27 (Springer, Berlin, 1939).

Exchange of Radioactive Silver with Silver Chloride Suspensions

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From exchange experiments on radioactive silver in solution with silver chloride in suspension, it can be shown that the silver ions in solution are constantly exchanging with the silver ions in the precipitate. This exchange is not limited to the surface of the crystals, but, probably by means of self-diffusion, is propagated into the precipitate. A steady radioactive state obtains when a homogeneous distribution of the radioactive silver throughout the whole system is reached. The time necessary to obtain a given fraction of complete exchange for a given amount of precipitate depends strongly on the area of the crystal surface.

THE exchange of silver ions in solution with silver chloride in suspension was studied, using radioactive silver as an exchange indicator. It was observed that the radioactive silver undergoes quite a rapid exchange with the precipitate, indicating that in all cases after sufficient time, a homogeneous distribution of the radioactive silver throughout the whole system can be obtained. The phenomenon was studied in detail with respect to the concentration of the silver in solution and the amount of precipitate used, with respect to the previous treatment of the silver chloride leading to different crystal sizes and consequently to different surfaces as determined by the dye adsorption method, and with respect to the effect of temperature. The influence of dyes previously adsorbed on the precipitates was also investigated. A possible interpretation of the observed curves in terms of diffusion and crystal size is given.