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Detection of the α-γ Iron Phase Transformation by Differential Thermal Conductivity Analysis

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A method of phase change detection in solids involving differential thermal conductivity analysis is described. This method is applied to the α-γ transformation of iron at various pressures up to 100 000 atm. This transformation temperature was found to drop continuously with increasing pressure down to 605°C at 100 000 atm.

A DIFFERENTIAL method of detecting phase changes in solids, involving measurement of the thermal conductivity changes accompanying the transformation, has been worked out recently. The method is entitled differential thermal conductivity analysis (DTCA), in contrast to differential thermal analysis (DTA), which involves changes in latent heat during a transformation.[1] This DTCA method has been used under super-pressure conditions, up to 100 000 atm, to determine the effect of pressure on the temperature at which the α-γ transformation in iron occurs. The method was planned especially for those systems where electrical conductivity measurements could not be used for detection, as in ceramic materials; this aspect is now being pursued with initial success with silica, boron oxide, and selenium; a report will be forthcoming in the future.

The DTCA method as first employed consisted in placing a temperature gradient across two parallel pieces of material: iron, whose transformation was to be investigated, and nickel, used as a reference material. The apparatus is illustrated schematically in Fig. 1. The temperature gradient in each of the pieces of metal would be linear and identical provided that each piece was homogeneous in itself, as shown in curve 1. If the iron transformed partly to a second phase of a different thermal conductivity than the initial phase, the temperature gradient through the iron would become nonlinear, as in curves 2 or 3. This departure from linearity was found to be easily detectable by placing two thermocouples, one at the mid-point of each piece of metal, bucking one thermocouple against the other, and thus measuring the difference in temperature ΔT. The sign of the ΔT would be positive or negative depending upon whether the new phase forming had a higher or a lower thermal conductivity, respectively, than the first phase.

The complete cell design, shown in Fig. 2, includes the following: insulating alumina disks above and below the iron and nickel strips; next, metal (nickel) disks above and below, to equalize the end temperatures of the metal strips; a lava insulator between the two metal strips; stacks of alumina pills at the cool end and lava pills at the hot end, these causing the temperature gradient in the metal strips by virtue of their (lava and alumina) differences in thermal conductivities; an alumina bushing surrounding the just-described center core; a second alumina bushing surrounding the first, between which was inserted four nickel strip heaters; an outer lava bushing enclosing the whole. This cell was placed in a super-pressure belt apparatus along with suitable gasketing material, described elsewhere.[2]

The ΔT signal was obtained across two platinum wires,

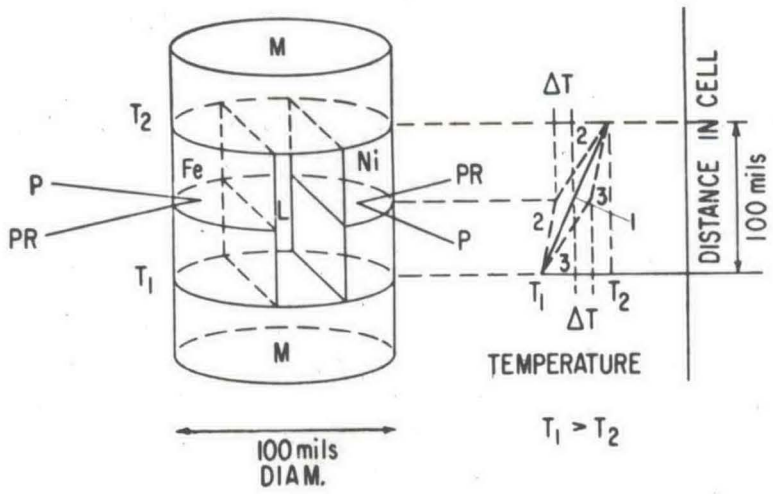

FIG. 1. Core design of DTCA cell. Thermocouples: P = platinum, PR = platinum −10% rhodium. ΔT, Read from the two P leads, with the two PR leads tied together. T, Read from "Fe" pair of P–PR leads. M = Metal pills for temperature equalization.

[1] J. Reilly and W. N. Rae, *Physico-Chemical Methods* (D. van Nostrand Company, Inc., Princeton, New Jersey, 1953), p. 221.
[2] H. Tracy Hall, Rev. Sci. Instr. 31, 125 (1960).