

THIS REPORT HAS BEEN DELIMITED
AND CLEARED FOR PUBLIC RELEASE
UNDER DOD DIRECTIVE 5200.20 AND
NO RESTRICTIONS ARE IMPOSED UPON
ITS USE AND DISCLOSURE.

DISTRIBUTION STATEMENT A

APPROVED FOR PUBLIC RELEASE;
DISTRIBUTION UNLIMITED.

Armed Services Technical Information Agency

Because of our limited supply, you are requested to return this copy WHEN IT HAS SERVED YOUR PURPOSE so that it may be made available to other requesters. Your cooperation will be appreciated.

AD 37778

NOTICE: WHEN GOVERNMENT OR OTHER DRAWINGS, SPECIFICATIONS OR OTHER DATA ARE USED FOR ANY PURPOSE OTHER THAN IN CONNECTION WITH A DEFINITELY RELATED GOVERNMENT PROCUREMENT OPERATION, THE U. S. GOVERNMENT THEREBY INCURS NO RESPONSIBILITY, NOR ANY OBLIGATION WHATSOEVER; AND THE FACT THAT THE GOVERNMENT MAY HAVE FORMULATED, FURNISHED, OR IN ANY WAY SUPPLIED THE SAID DRAWINGS, SPECIFICATIONS, OR OTHER DATA IS NOT TO BE REGARDED BY IMPLICATION OR OTHERWISE AS IN ANY MANNER LICENSING THE HOLDER OR ANY OTHER PERSON OR CORPORATION, OR CONVEYING ANY RIGHTS OR PERMISSION TO MANUFACTURE, USE OR SELL ANY PATENTED INVENTION THAT MAY IN ANY WAY BE RELATED THERETO.

**Reproduced by
DOCUMENT SERVICE CENTER
KNOTT BUILDING, DAYTON, 2, OHIO**

UNCLASSIFIED

AD No. 32778

ASTIA FILE COPY

Ninth Progress Report
Contract Nonr 266(07)
THE FUNDAMENTALS OF THE TRANSITION
TEMPERATURE PHENOMENON IN STEEL
J. O. Brittain and M. Gensamer
1 Oct. 1953 - 31 Dec. 1953
CU-9-53-Nonr 266(07)-Met.

Ninth Progress Report
Contract Nonr 266(07)

FUNDAMENTALS OF THE
TRANSITION TEMPERATURE PHENOMENON
IN STEEL

J. O. Brittain and M. Gensamer

1 October 1953 - 31 December 1953

COLUMBIA UNIVERSITY
School of Mines
New York 27, N. Y.

19 February 1954

Dr. Julius Harwood, T3-2710
Office of Naval Research
Washington 25, D. C.

Dear Dr. Harwood:

This letter constitutes the ninth report of progress for the period October 1, 1953 to December 31, 1953 on Contract Nonr 266(O7), entitled, "Fundamentals of the Transition Temperature Phenomenon in Steel."

During the period covered by this report the study of the effect of hydrogen in iron has been continued with the construction of the out-gassing apparatus and the adaptation of the internal friction machine to operate at temperatures below 77°K. A test program has been laid out to study the strain-aging behavior of iron-hydrogen alloys. Further attempts have been made to prepare a high purity iron which can be fabricated at 77°K. The study of the effect of microstructure, strain rate, and thermal and mechanical history on a 1020 steel and a vacuum melted electrolytic iron has been resumed with the modification of the hydrogen cryostat to reduce low temperature freezing of the gas lines.

Effect of Thermal and Mechanical History on the Transition Temperature:

In the period covered by this progress report, some difficulties were encountered with the hydrogen cryostat. The system has been modified to minimize freezing of either water, or oxygen, or both, in the system by the introduction of a larger drying tube in the system. In addition the expansion valve has been modified to decrease the bending of the valve rod in event the valve freezes in place so that a greater force can be exerted on the valve junction to open the valve.

The Instron testing machine has been installed in the laboratory and equipped with pull rods and specimen grips. The unit is being checked for elastic extension both in the unit and in the specimen adapters.

Preparation of Ductile Iron:

The experiments on the low temperature fabrication of vacuum remelted NRC vacuum melted electrolytic iron has continued. Ingot 7 was melted with a molybdenum radiation shield

until molten at which time the shield was removed and liquid nitrogen added to the freeze out. The charge of 200 grams of iron melted in about four minutes and was held molten for one minute. The directional solidification technique required about 10 minutes to solidify the ingot and final freezing occurred in a vacuum of about 10^{-5} mm of Hg.

Inasmuch as the previous ingots which had failed when rolled at 77°K had fractured in a predominantly intercrystalline manner, an experiment was set up to study the influence of rate of cooling from 920°C upon the ductility of the ingot when rolled at 77°K. The hypothesis was that rapid cooling from the austenite range would modify the grain boundary reaction to such an extent as to decrease the tendency to intercrystalline failure. Thus, this ingot was annealed in a vacuum of approximately 40 microns at 920°C and quenched into diffusion pump oil. When this ingot was rolled at 77°K, it showed somewhat better ductility than those ingots rolled directly from the cast condition but failed at a rather small reduction in area. Two more ingots which had been made under identical melting conditions as ingot 7 were rolled to 20% reduction and the other to 10% reduction at 195°K. It was planned to continue rolling at 77°K but a failure of a gear in the rolling mill has delayed this experiment.

The Study of the Effect of Hydrogen in Iron:

As a consequence of the recently reported findings of an internal friction peak at about 100°K in a hydrogen impregnated 1020 steel (1), a strain-aging experiment has been devised to test the hypothesis of the similarity of the role of hydrogen and carbon and nitrogen in iron. It is felt that the demonstration of a strain-aging behavior at 77°K in a 1020-hydrogen steel would amply confirm the predicted similarity of the role of hydrogen, carbon, and nitrogen in iron. It has been learned in a private communication that a yield point definitely attributed to hydrogen has been found at 77°K. Also Smallman et al (2) have demonstrated a yield point in aluminum when loaded with hydrogen, prestrained at 88°K and aged at 291°K for various lengths of time and restraining at 88°K.

Specimens for this experiment will be made from normalized SAE 1020 steel which will be annealed in hydrogen at about 875°K under a pressure of about 60 atmospheres. This will be followed by prestraining at 77°K followed by

aging at about 100°K and testing at 77°K. The aging temperature range is low enough to preclude aging effects due to either carbon or nitrogen.

Studies of the internal friction of the 1020 steel at low temperatures are being continued. The lowest temperature that could be reached with the present apparatus using liquid nitrogen was about 67°K. Preliminary studies in this temperature region showed that the internal friction of the 1020 steel as annealed and as hydrogen impregnated did not decrease continuously below the 100°K peak but tends to increase again on cooling to about 72°K, thus indicating the possibility of the presence of another internal friction peak. The magnitude of the internal friction peak was of course different for the two cases, being greater for the hydrogen impregnated specimen. Measurements at still lower temperatures will be continued as soon as the internal friction apparatus has been modified to use liquid helium or liquid hydrogen as the cooling medium.

Studies of the internal friction of molybdenum from room temperature to 77°K indicate the presence of two or possibly three peaks: one at about 300°K (reported previously), a second at about 180°K, and possibly a third at about 130°K. Further studies of the nature of these peaks are planned.

Although the construction of an entirely new solid state degassing system had been previously decided upon, it was hoped that some preliminary information concerning the optimum out-gassing conditions might be derived through the use of an existing system, consisting of a mechanical fore-pump and umbrella-type mercury diffusion pump with a pumping capacity of about 2 liters per second at a vacuum of 10^{-6} mm Hg; but operational difficulties encountered during long time annealing in this system lead to it being abandoned in favor of the designing and constructing of a two component system: an all-glass vacuum melting unit and an ultra-high vacuum solid state degassing system.

The all-glass vacuum melting furnace has been designed to permit rapid removal of gas in relation to the mass of metal melted, while maintaining a pressure of about 10^{-7} mm Hg throughout the melting operation. The furnace will be evacuated by a three-stage umbrella-type mercury diffusion pump with a pumping speed of 80 liters per second at 10^{-6} mm Hg. The melt will consist of 0.2 to 0.3 cubic inch of iron that had been previously vacuum melted. Zirconia and alumina crucibles will be used to contain the melt. A furnace head

has been designed with a liquid nitrogen freeze-out near the surface of the melt. The furnace head is sealed by a short mercury column which also provides an easy method of breaking vacuum without breaking glass. A worm gear raising and lowering assembly for the induction coil will be provided to minimize the formation of a pipe in the ingot. Ingots will be cold worked 75% by swaging, machined to the desired specimen dimension, and treated further in the solid state degassing system.

The ultra-high vacuum system for solid state degassing has to a large extent been patterned after a system described by D. Alpert in a recent publication (3). The pumping action below pressures of the order of 10^{-7} mm Hg, as well as the measurement of these pressures is provided by the Bayard-Alpert ionization gage. This gage operates in a manner similar to conventional ionization gages, except that the ion collector intercepts only a very small fraction of the X-rays produced at the grid. It is this feature that permits the measurement of vacua better than 10^{-7} mm of Hg, since the conventional ionization gage becomes inaccurate at extremely low pressures due to interference by the above mentioned X-rays. In the Bayard-Alpert gage the filament is outside the cylindrical grid while the ion collector, which is a fine wire, is suspended within the grid. Gas (especially H_2) disassociates as it strikes the tungsten filament, which normally operates at $2300^\circ K$. The ions are attracted to the negatively charged surfaces of the gage, where they are trapped. A chemical pumping action (adsorption) exists as well, and the rate of pressure reduction due to both actions has been calculated (3).

Another essential component of the ultra-high vacuum system is a mechanical valve. The valves have been designed and built by the Westinghouse Research Laboratory for use in the isolation of parts of vacuum systems at 10^{-9} mm Hg from pressures on the order of 10^{-7} mm Hg for a period of over 30 days with no observable increase in pressure on the ultra-high vacuum side. The needle action of the valve is provided by a differential screw driving mechanism.

The entire system has been arranged to permit bake-out at $450^\circ C$, and is sufficiently flexible to permit easy replacement of components and changes. The electronic components were designed to enable measurement by both conventional ionization gages and Bayard-Alpert gages. In order to insure an adequate fore pressure for the ion pump (10^{-7} mm Hg) it is necessary to maintain a level of liquid nitrogen in the refrigerated trap between the mercury

diffusion pump and the first mechanical vacuum valve. An arrangement has been devised using a floater activating a timing relay which in turn opens a solenoid valve to permit nitrogen gas to pump liquid nitrogen into the cold trap.

Several heating and cooling cycles are presently being contemplated to facilitate the removal of hydrogen in ultra-high vacuum.

1. Induction heating of specimens at a temperature just below the α - γ transformation, taking advantage of the most favorable diffusion rate (D_{α} 800°C $> 2.7 \times 10^{-4}$ cm² sec⁻¹). Such treatment would also be accompanied by grain growth, and hence some grain boundary migration.

2. Oscillation between α and γ phases, producing more grain boundary migration.

3. Direct passage of current through specimen. With such a scheme it is deemed possible that a preferential migration of protons (since in the Fe lattice some hydrogen exists in the form of protons) could be accomplished.

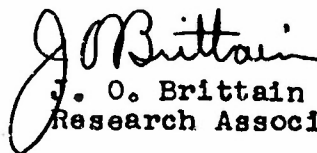
Future Work:

Work in the next quarterly period will continue along the lines indicated in this report.

References:

- (1) Sixth Progress Report on Contract Nonr 266(O7), April, 1953. Lo-ching Chang and M. Gensamer, Acta Metallurgica 1, (1953), 483.
- (2) Smallman, R. E., Williamson, G. K. and Ardley, G., Acta Metallurgica 1, (1953), 126.
- (3) Westinghouse Research Laboratories, East Pittsburgh, Pennsylvania, Scientific Paper #1695.

Respectfully submitted,


J. O. Brittain
Research Associate

ORIGINAL SIGNED BY
M. GENSAMER

M. Gensamer
Professor of Metallurgy

JOB:mb

Armed Services Technical Information Agency

Because of our limited supply, you are requested to return this copy WHEN IT HAS SERVED YOUR PURPOSE so that it may be made available to other requesters. Your cooperation will be appreciated.

AD

37778

NOTICE: WHEN GOVERNMENT OR OTHER DRAWINGS, SPECIFICATIONS OR OTHER DATA ARE USED FOR ANY PURPOSE OTHER THAN IN CONNECTION WITH A DEFINITELY RELATED GOVERNMENT PROCUREMENT OPERATION, THE U. S. GOVERNMENT THEREBY INCURS NO RESPONSIBILITY, NOR ANY OBLIGATION WHATSOEVER; AND THE FACT THAT THE GOVERNMENT MAY HAVE FORMULATED, FURNISHED, OR IN ANY WAY SUPPLIED THE SAID DRAWINGS, SPECIFICATIONS, OR OTHER DATA IS NOT TO BE REGARDED BY IMPLICATION OR OTHERWISE AS IN ANY MANNER LICENSING THE HOLDER OR ANY OTHER PERSON OR CORPORATION, OR CONVEYING ANY RIGHTS OR PERMISSION TO MANUFACTURE, USE OR SELL ANY PATENTED INVENTION THAT MAY IN ANY WAY BE RELATED THERETO.

Reproduced by
DOCUMENT SERVICE CENTER
KNOTT BUILDING, DAYTON, 2, OHIO

UNCLASSIFIED