Heat Capacity of Hastelloy Alloy X
at Temperatures between 37° C. and 1100° C.

RICHARD R. THAIS and JAMES P. KOHN
Department of Chemical Engineering, University of Notre Dame, Notre Dame, Ind.

The heat capacity of Hastelloy Alloy X was determined by a drop method employing a water calorimeter over the temperature range from 37° to 1100° C. The estimated maximum error is 3% over this temperature range.

THE THERMAL PROPERTIES of alloys having high tensile strength and good corrosion resistance at elevated temperatures constitute important fundamental data for the accurate design of structural components for high temperature uses in space, nuclear, and combustion technology. Although the thermal properties of certain metallic alloys have been reported recently (1, 4), the heat capacity of Hastelloy Alloy X at elevated temperatures has not been published previously.

EXPERIMENTAL

The calorimeter was constructed from a standard small cylindrical Dewar flask which was fitted with a plastic top. The top was machined to accommodate a small, thin-walled cylindrical brass container which served as a distilled water reservoir. The outside of the container was chromium-plated to reduce radiation losses and to obtain a reproducible surface so that heat losses would be independent of surface effects. Inside the container, a copper stirring disk was attached to a thin stiff wire, and the thermocouple was also attached to the disk. Thorough mixing of the water in the reservoir was obtained by raising and lowering the disk at the rate of about two cycles per second.

The furnace used to heat the sample was a well-insulated, nichrome-wound quartz tube mounted directly above the calorimeter and about 18 inches from the top of it.

The enthalpy of a small sample of Alloy X was measured by rapidly submerging the heated sample, suspended on a nichrome fuse wire, into the brass calorimeter containing an accurately known amount of distilled water. The size of the sample was between 15 and 30 grams, the smaller sample being used for high temperatures. The water in the brass calorimeter weighed between 20 and 90 grams, depending on the size of the sample, and the temperature of the sample before it was dropped. The amount of water to be used was determined from the following criteria:

The change in the water temperature should be great enough to minimize errors in measuring this temperature change.

The maximum bulk temperature of the water must not exceed 50° C. because above this temperature there is an appreciable heat loss caused by vaporization of the water.

The amount of water must be great enough so that weighing errors are negligible.

The temperature of the water was measured as a function of time by means of a single junction, copper-constantan thermocouple which was calibrated using the boiling points of distilled water and pure mercury as standards. The voltage of the thermocouple was measured with a precision potentiometer with the melting point of ice as the cold-junction temperature. The estimated accuracy of the temperature reading was 0.05° C.

The plot of the temperature of the water vs. time was extrapolated to the time when the sample was lowered into the water. The error in this technique was calculated to be less than 0.5%.

Since the brass calorimeter had very thin walls (0.015 inch), a low heat capacity, and the mass of the stirring disk and submerged portion of the thermocouple and stirring wire is very small, the vessel itself absorbed less than 10% of the heat transferred to the calorimeter. This made it impractical to obtain an accurate experimental calibration of the heat capacity of the tube. The portion of the calorimeter above the water interface was treated as a slab, and the steady state heat conduction equation with convection heat loss from both inside and outside was solved with the slab having one end at a constant temperature and the other end insulated. The integral mean temperature change was obtained, and this was used to obtain the heat content of the section of the tube above the interface. The portion of the tube below the interface, the stirring disk, and the submerged portion of the wires were considered as undergoing the same temperature change as the water in the calorimeter. The estimated error in using this calculated calorimeter calibration as the actual tube calibration was less than 0.5%.

The maximum heat loss caused by radiation and convection in lowering the sample to the calorimeter is 0.7% The tube was filled to different levels with water at different temperatures to determine the heat flux from the tube as a function of temperature difference and height of water in the tube.

Hot samples were rapidly lowered into the full tube, and the difference in the weights before and after the drop gave an estimate of the amount of water lost by vaporization. Using a brass sample much larger than those used in a regular run, a maximum loss of about 0.7% of the total heat of the sample was found for an initial sample temperature of 900° C. The Alloy X samples were tried to 1100° C., but an

amount of water equivalent to $< 0.5\%$ of the heat of the sample was lost. This small loss can be explained by the fact that film boiling occurred in these cases, and the film around the sample reduced the rate of formation of vapor as the sample passed through the water interface. Once the sample was submerged, all the steam condensed in the cool water before it could escape to the interface. No doubt, part of the steam condensed on the wall of the brass tube, but it was thought that this small amount could be considered negligible.

The samples of Hastelloy Alloy X were furnished by the Haynes Sellite Division of the Union Carbide Corp. The samples were stated to come from "heats" having the following nominal composition ranges

	Wt. %		
Carbon	0.05	–	0.15
Chromium	20.50	–	23.00
Cobalt	0.50	–	2.50
Iron	17.00	–	20.00
Manganese	1.00	max.	
Molybdenum	8.00	–	10.00
Nickel	Remainder		
Silicon	1.00	max.	
Tungsten	0.20	–	1.00

RESULTS

Fifty-five experimental points were determined at temperatures between 37.6° and 1095° C. (Only 49 points are shown on the graph because some were so closely reproduced that the two circles could not be distinguished on a plot this small.) On the basis of the low temperature runs, the enthalpy curve was extrapolated to 0° C. at which point the enthalpy was defined as zero. After attempts to fit the data to the equation suggested by Kelley (2) and a standard least-square polynomial fit using an IBM 1620 computer proved unsatisfactory, a curve fit based on a trial and error technique yielded the following equation.

$$H_t - H_o = 0.1026T + 2.20 \times 10^{-5}T^2 + 2.30 \times 10^{-18}T^6 \quad (1)$$
T in ° C.
H in cal./gram
H_o = enthalpy at 0° C.

This equation for the enthalpy of Hastelloy Alloy X between 0° and 1100° C. provides a fit to the experimental data which give a standard deviation of ± 0.70 cal./gram or about 1.25%, with the maximum deviation being less than 3%. This equation fits the data within the experimental precision of the apparatus.

The experimental data and the plot of Equation 1 are shown on Figure 1. The equation and the experimental points appear in good agreement, except for the four data points near 900° C. These four points have positive deviations which appear to be too ordered to be random errors. They could possibly indicate some sort of a transformation in the alloy. It was not possible from this study to ascertain if there were a transformation or not.

Since Equation 1 does not contain any terms of negative sign, it is reasonable to assume that the first derivative of
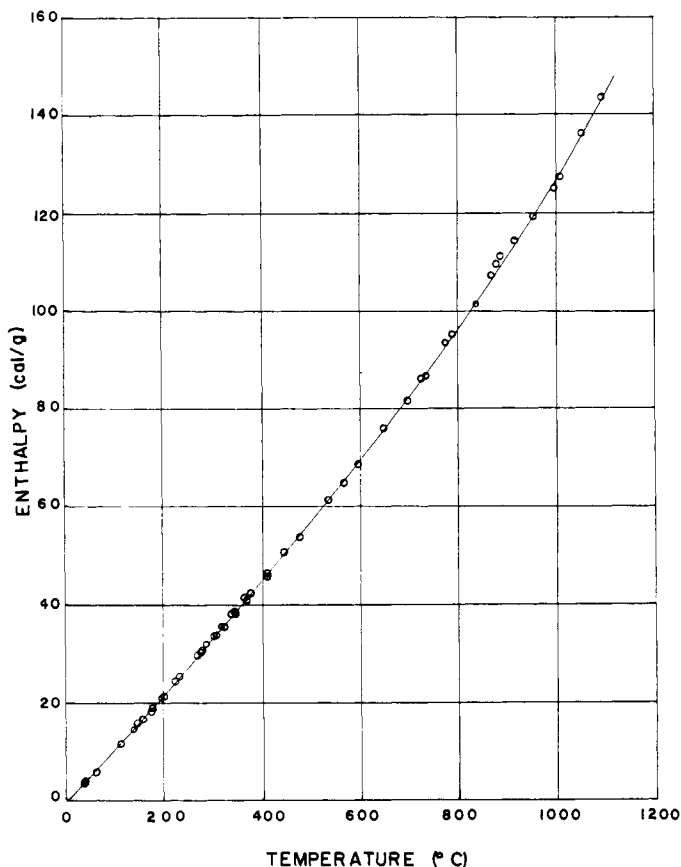

Figure 1. Enthalpy of Hastelloy Alloy X vs. temperature

the equation with respect to temperature should yield an accurate equation for the heat capacity. The resulting heat capacity equation is:

$$C_P = 0.1026 + 4.40 \times 10^{-5}T + 1.38 \times 10^{-17}T^5 \quad (2)$$

Equation 2 gives the value of the capacity of Hastelloy Alloy X within 2.0% of the values determined in an unpublished study by Southern Research Institute (3) at temperatures up to 800° C.

LITERATURE CITED

(1) Goldsmith, A., Waterman, T., Hirschhorn, H., "Handbook of Thermophysical Properties of Solid Materials," Vol. 2, Macmillan, New York, 1961.
(2) Kelley, K. K., Bureau of Mines Bul., No. 476, 1949.
(3) Pears, C.D., Southern Research Institute, Birmingham, Ala., private communication, Nov. 1960.
(4) Thermophysical Properties Research Center, TPRC Data Books, 1, 3, Purdue University, June 1962.