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THERMOGRAVIMETRIC STUDY OF THE REDUCTION OF OXIDES OF NICKEL AND CHROMIUM

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16. Abstract

The controlled hydrogen, carbon, and hydrogen-carbon reductions of the oxides of nickel and chromium were evaluated by thermogravimetric means. The materials studied were nickel (nickelous) oxide (NiO) and chromic sesquioxide (Cr_2O_3), mixed NiO-Cr_2O_3, and oxidized nickel - 20-percent chromium (Ni-20Cr). NiO was effectively reduced by all three atmospheres, Cr_2O_3 only by hydrogen-carbon, NiO-Cr_2O_3 by hydrogen and hydrogen-carbon, and oxidized Ni-20Cr by hydrogen and hydrogen-carbon and to a considerable extent by carbon alone. The results suggest that the presence of nickel promotes the reduction of Cr_2O_3. However, no definite explanation could be reached for the effectiveness of the hydrogen-carbon reduction of Cr_2O_3.

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THERMOGRAVIMETRIC STUDY OF THE REDUCTION OF OXIDES OF NICKEL AND CHROMIUM

by Thomas P. Herbell

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SUMMARY

This study was carried out to determine the effect of controlled hydrogen, carbon, and hydrogen-carbon reductions of the major contaminant oxides present in nickel chromium alloys prepared by powder metallurgy. The materials studied were nickel (nichelous) oxide (NiO), chromic sesquioxide (Cr_2O_3), mixed NiO-Cr_2O_3, and oxidized nickel - 20-percent chromium (Ni-20Cr). The reductions were carried out in a TGA (thermogravimetric analysis) apparatus. The sample heating in all cases was linear with time and at a rate of 3° C per minute from ambient to 1200° C. The reducing agents were hydrogen, carbon, and a combination of the two. Reduction was evaluated by weight loss data and by X-ray analysis of the reduction residue.

Hydrogen, carbon, and hydrogen-carbon were all effective in reducing NiO. However, complete reduction was achieved at the lowest temperature with dry hydrogen. Only hydrogen-carbon was effective in reducing Cr_2O_3. The mixed oxides were reduced by hydrogen and hydrogen-carbon. The oxidized Ni-20Cr was reduced by hydrogen and hydrogen-carbon and to a considerable extent by carbon alone.

The results suggest that the presence of nickel promotes the reduction of Cr_2O_3. However, no definite explanation could be reached to explain the effectiveness of the hydrogen-carbon reduction of Cr_2O_3.

INTRODUCTION

For some time NASA has been involved in research programs aimed toward the production of dispersion-strengthened alloys containing chromium. Dispersion-strengthened alloys based on nickel - 20-percent chromium (Ni-20Cr) have potential for use in a variety of high-temperature applications such as components of advanced air-breathing engines and space vehicles. Work carried out at the Lewis Research Center of NASA has clearly established that the successful processing of high-temperature, dispersion-strengthened materials requires that some types of contamination be held to a minimum (ref. 1). Matrix oxides (oxides of the metallic components of a dispersion-strengthened alloy), particularly those with a high negative free energy of formation such as Cr_2O_3, appear to be deleterious. These matrix oxides must be reduced or eliminated if stable, high-temperature materials are to be produced. These oxides restrict sintering (ref. 2), and there are implications that they also lower the stability of the deliberately added oxide dispersoid (refs. 3 and 4), thus lowering its effectiveness as a strengthener. There are a number of approaches to the removal of Cr_2O_3, such as reduction by hydrogen (ref. 5), by carbon (ref. 6), and by the hydrogen halides (ref. 7).

The objective of this study was to evaluate the controlled hydrogen, carbon, and hydrogen-carbon reduction of oxides in Ni-20Cr. A TGA (thermogravimetric analysis) apparatus designed and constructed at NASA Lewis and described in reference 8 was used in this study. The materials chosen for evaluation were nickel (nickelous) oxide (NiO), chromic sesquioxide (Cr_2O_3), mixed NiO-Cr_2O_3, and deliberately oxidized Ni-20Cr powder. The NiO-Cr_2O_3 was included to generate baseline data for alloy systems where more than one matrix oxide might be encountered. The reducing agents were dry hydrogen, carbon, and a combination of the two. Twelve controlled-reduction experiments were carried out in duplicate. The heating rate was linear with time and at a rate of 3° C per minute. Reductions were evaluated by weight loss data and by X-ray analysis of the residue from each reduction experiment.

MATERIALS, APPARATUS, AND PROCEDURE

Materials

The materials used in this study were Fisher "certified" reagent-grade NiO and Cr_2O_3, spectrographic-grade lampblack, ultra-high-purity hydrogen, and research-grade helium. The moisture level of the hydrogen and helium was reduced to less than 0.03 ppm by the procedure described in reference 8.

The deliberately oxidized Ni-20Cr powder was prepared by firing inert gas atomized Ni-20Cr powder in an alumina crucible for 6 hours in pure oxygen at 1100° C. After firing, this material consisted essentially of NiO and the spinel $NiCr_2O_4$. The total analyzed oxygen content of the oxidized material was 22.16 percent.

The oxide and oxide-plus-carbon mixtures necessary for the reduction experiments were made by dispersing weighed amounts of the constituents in 200-proof alcohol, followed by air drying at 100° C. Approximately 10 grams of each blend were prepared. The amount of carbon added to the blends containing carbon was just sufficient for the stoichiometric conversion of the oxygen present to carbon dioxide (CO_2). Selection of the carbon addition was based on preliminary data and is not meant to infer that the reduction procedes exclusively by the formation of CO_2. For example, in a preliminary unpublished study by the author it was found that, when sufficient carbon was added for the stoichiometric conversion of the oxygen to CO, the residue contained metal carbides. With the carbon addition based on the formation of CO_2, no carbides were detected and the oxygen removal reaction appeared to go to completion.

Apparatus

The essential features of the TGA apparatus used have been described previously (ref. 8). A schematic drawing of the balance-and-reaction-tube assembly is shown in figure 1. Several modifications to the apparatus were required to accommodate the use of carbonaceous atmospheres and the higher temperatures required for the reduction of Cr_2O_3. Specifically, the reaction-gas-inlet and thermocouple-protection tubes were changed from stainless steel to quartz. The quartz crucible was changed to triple recrystallized alumina. The original Nichrome-wound furnace was replaced with a Kanthal-wound furnace capable of temperatures up to 1300° C. A final modification was the incorporation of the automatic shutdown procedure described in reference 9 which automatically turned off the entire electrical system at the end of the temperature program.

Procedure

A sample to be reduced was weighed directly into the crucible on an auxiliary balance located outside the TGA system. Weighing was done to the nearest 0.1 milligram, which was the sensitivity of the balance used. The weight of material used was predetermined either from formula weight or from chemical analysis to give a weight loss close to 10 milligrams upon complete reduction to the metal or alloy. Placing exactly the right amount of material - to the nearest 0.1 milligram - in the crucible was difficult. Therefore, in some instances slightly less than the desired amount of sample was used. To compensate for this weight factor, all data curves were subsequently normalized for a total weight loss of exactly 10 milligrams for complete removal of the oxygen, and where present, the deliberately added carbon; that is, with carbon present the 10-milligram weight loss represents 7.3 milligrams of oxygen and 2.7 milligrams of carbon.

The crucible containing the sample was attached to the quartz support fiber which was suspended from the balance. After the tare weights were adjusted, the system was evacuated to remove air and moisture and then backfilled with the desired atmosphere, that is, hydrogen for the hydrogen and hydrogen-carbon reductions and helium for the carbon reduction. When equilibrium was established at the desired flow rate of 150 cubic centimeters per minute, the heating program was started.

Before the experimental runs were started, a blank crucible (buoyancy calibration) run was carried out to correct for changes in density of the flowing gases and other fluctuations that occur as a function of temperature. Blank crucible runs were carried out in both hydrogen and helium. In the blank crucible runs, as well as in all the experimental runs, the purge gas was 50 cubic centimeters per minute of helium. A separate temperature-against-time curve was obtained for each experimental run to verify the linearity of the temperature program. The heating rate was linear in all cases and at a constant rate of 3° C per minute.

RESULTS AND DISCUSSION

Table I summarizes the sample materials, the reducing conditions, and the percent of the calculated weight loss based on the reduction of the sample to metal for each run. The X-ray analysis results for the final residue from each run are included as a footnote to this table.

Weight-loss data presented in table I were obtained from direct measurement of the curves for weight loss as a function of temperature. TGA weight-loss curves for the hydrogen, carbon, and hydrogen-carbon reductions of NiO, Cr_2O_3, mixed NiO-Cr_2O_3, and oxidized Ni-20Cr are shown in figures 2 to 5. Each of the curves presented in these figures has been corrected for the effect of buoyancy. Where necessary, each experimental curve was normalized for a 10-milligram weight loss for complete reduction to metal.

In referring to table I, note that in some instances reductions of greater than 100 percent were achieved. In all cases the values reported appear to be within the limit of accuracy of the system. The normal accuracy of the system to a temperature of 1000° C is ±1 percent. At 1200° C, however, the increased uncertainty in the buoyancy correction decreases the limit of accuracy to the order of ±2. 5 percent.

Reduction of Nickel (Nickelous) Oxide

As can be seen in table I, the reduction of NiO went to 100-percent completion in hydrogen, 98. 2-percent completion in hydrogen-carbon, and 95. 4-percent completion with carbon alone. The residue from the hydrogen and hydrogen-carbon reductions was metallic grey in color, and the X-ray analysis indicated only pure nickel to be present. With carbon alone, the residue was black in color and the X-ray analysis indicated that it was essentially nickel with a trace of NiO.

In order to gain a better understanding of what is happening in the reduction of NiO, it is necessary to refer to figure 2. In this figure we see that with hydrogen the reduction occurred over a very narrow temperature range, from about 235° to 310° C. When carbon is added to the sample and reduction is then carried out in a hydrogen atmosphere, a weight loss of 6.9 milligrams occurred in the temperature range 225° to 285° C. If it is assumed that the NiO and carbon mixture was uniform and that the original weight loss was caused only by the removal of oxygen, then the weight loss should have been 7.3 milligrams. The difference between a loss of 7.3 milligrams and the observed loss of 6.9 milligrams could be the result of a slight inhomogeneity in the NiO-carbon blend. Presumably, the remaining slow weight loss to a temperature in the neighborhood of 1100° C was caused by the removal of the carbon present in the blend - probably as methane (CH_4) (ref. 10).

Reduction with carbon produced a rapid weight loss of about 2 milligrams; however, this did not occur until a temperature of 670° C was reached. The initial rapid loss was followed by a slower loss of an additional 2 milligrams between 670° and 800° C and then a final loss between 800° and 1150° C, yielding a total percentage reduction of 95.4. This particular reduction terminated at 1150° C and perhaps would have been more complete had the maximum temperature of 1200° C been achieved. Interpretation of the three distinct reaction steps in the curve is beyond the scope of the present investigation.

Reduction of Chromic Sesquioxide

Table I reveals that Cr_2O_3 was only slightly reduced with either hydrogen or carbon alone, the percent reductions being 30.1 and 29.0, respectively. When hydrogen and carbon were used in combination, however, the reduction went to 97.2-percent completion. The residue from the hydrogen reduction was green, and the X-ray analysis indicated the presence of both chromium and Cr_2O_3. With carbon the residue was black in color and only Cr_2O_3 was detected. Reduction with a combination of hydrogen and carbon produced a grey residue which was analyzed by X-ray diffraction as pure chromium.

The weight-loss curves for the reduction of Cr_2O_3 are shown in figure 3. It is readily apparent from this figure that hydrogen or carbon alone under the conditions of this study were not effective in reducing Cr_2O_3. With hydrogen, very little weight was lost until a temperature of approximately 1000° C was attained. Between 1000° and 1200° C, however, there was a relatively rapid loss of about 2.5 milligrams. The use of carbon produced a similar slow weight loss which continued all the way to about 1175° C. At this temperature, there was a rapid loss of the order of 2 milligrams. The total weight loss with carbon was only 2.9 milligrams.

The combination of hydrogen and carbon appears to be effective in reducing Cr_2O_3. Not only did the reduction go almost to completion, but it also started at a lower temperature than for either hydrogen or carbon alone. The weight loss began at about 900° C and continued at a relatively rapid rate to the maximum temperature used - 1200° C.

The reason for the observed effectiveness of the hydrogen-carbon reduction is not readily understood. Perhaps it involves the formation of some hydrocarbon vapor which accomplishes the reduction. In this respect, however, it has been observed by the author that pure methane (CH_4) does not reduce Cr_2O_3, but rather the gas converts the oxide to chromium carbide.

Hopkins (ref. 11) states that Cr_2O_3 can be reduced by carbon, provided vacuum or a dilute inert gas are used. And Jones (ref. 12) indicates that the combination of hydrogen and carbon is particularly effective. Further study is required, however, to determine the reason for the effectiveness of the hydrogen-carbon reduction.

Reduction of Mixed Nickel (Nickelous) Oxide and Chromic Sesquioxide

Hydrogen or hydrogen plus carbon appear to be effective reductants for 80-20 (based on metal content) mixtures of NiO and Cr_2O_3. From table I, note that weight losses of 97.8 and 102.3 percent were achieved with hydrogen and hydrogen-carbon, respectively. The residue from the hydrogen and hydrogen-carbon reduction was metallic silver in color, and the X-ray analysis indicated only the presence of a nickel solid solution. Based on both X-ray analysis and spectrographic analysis, the chromium content of this solid solution was essentially the same as for the starting mixture, that is, 20-percent chromium. Carbon reduction yielded a green residue which contained NiO, $NiCr_2O_4$, and a nickel solid solution with slightly less than 20-percent chromium.

Dry hydrogen was obviously very effective in reducing the mixed NiO-Cr_2O_3 and, as will be described in the next section, was equally effective in the reduction of oxidized Ni-20Cr. These results, combined with the lack of reduction of pure Cr_2O_3 in the same atmosphere, require explanation. In order to rationalize this behavior, we must take the following points into consideration:

(1) Meyer (ref. 13) observed that iron-Cr_2O_3 mixtures could be completely reduced in hydrogen at 1350° C to produce an iron - 19.1-percent-chromium alloy.

(2) Baukloh and Henke (ref. 14) were able to completely reduce Cr_2O_3 in hydrogen at 1400° C; however, in the presence of iron, complete reduction occurred at 1200° C.

(3) Barshchevskaya and Radomysel'skii (ref. 15) noted a similar improvement in the hydrogen reduction of Cr_2O_3 in mixtures and compounds based on iron oxide and chromium oxide.

Apparently Cr_2O_3 can be reduced in the presence of iron because the freshly reduced chromium is removed from the reaction by alloying with the iron. This is aided by the fact that the diffusion rate of chromium into iron is greater than the diffusion rate of iron into chromium. A similar situation exists with the nickel-chromium system. Thus, it is reasonable to postulate that the freshly reduced chromium forms a solid solution with nickel and, provided the dewpoint of the surrounding atmosphere is low enough to prevent reoxidation, the reaction continues until all the chromium oxide is reduced and alloyed with the nickel.

The reduction curves shown in figure 4 indicate that hydrogen and hydrogen-carbon were approximately equally effective in reducing the oxide mixture. The major weight loss occurred in the temperature range 230° to 296° C. At this point the hydrogen-reduced material had lost almost 6.8 milligrams, and the hydrogen-carbon-reduced material almost 6.3 milligrams. Following the initial rapid weight loss, the hydrogen-reduced material exhibited a weight plateau all the way to about 700° C. The remaining weight loss occurred between 700° and 1100° C and was followed by a plateau between 1100° and 1200° C. With hydrogen plus carbon, there was also a plateau after the original rapid weight loss; however, this terminated at about 525° C. The remaining weight loss was in two stages separated by a slight plateau at about 600° C and was complete at slightly above 1000° C.

Reduction with carbon alone was markedly different from the hydrogen or hydrogen-carbon reduction. Figure 4 shows that no weight loss occurred until a temperature of greater than 700° C was achieved. At this temperature there was a rapid loss of slightly more than 2 milligrams. The remaining loss, to a total of 3.4 milligrams, was very gradual and ended at approximately 1150° C.

Reduction of Ni-20Cr

Hydrogen, carbon, and hydrogen-carbon all appeared effective in reducing oxidized Ni-20Cr. As shown in table I, the hydrogen reduction went to 100-percent completion, the hydrogen-carbon reduction to 103-percent completion, and the carbon reduction to 72-percent completion. The residue from the hydrogen and hydrogen-carbon reductions was metallic silver in color, and the X-ray analysis indicated only a nickel solid solution to be present. Based on both X-ray diffraction and spectrographic analysis, the chromium content of this solid solution was essentially the same as for the starting alloy, that is, 20-percent chromium. The residue from the carbon reduction was black in color and contained a nickel solid solution and Cr_2O_3. No carbides were detected.

The reduction curves shown in figure 5 reveal that the hydrogen and hydrogen-carbon reductions were generally very similar. Both weight losses started at about 350° C and continued to about 1150° C. The primary difference between the two reductions was that for any given temperature the weight loss with hydrogen was greater than with hydrogen-carbon. The most probable reason for this difference is the fact that carbon does not react appreciably with the oxides until a temperature of the order of 1000° C is reached.

At temperatures greater than 1000° C the two weight-loss curves rapidly approach each other.

Oxidized Ni-20Cr reduced with carbon did not begin to lose weight until much higher temperatures were reached. Reduction was characterized by a slow weight loss, starting at about 700° C. The main weight loss occurred rapidly in the temperature range 1000° to 1200° C. From the appearance of the curve, it is quite probable that this reduction would have gone more to completion at slightly higher temperatures or if held isothermally at 1200° C.

CONCLUDING REMARKS

The significance of the results of this study lies in being able to utilize the reduction data obtained to select reasonable temperature ranges and reducing atmospheres for use in batch reductions of metal powders which unavoidably contain the oxides studied in this investigation. It is well known that even carefully controlled powder preparation procedures result in small amounts of oxygen contamination. Furthermore, various comminution and handling procedures required in the production of high-temperature, dispersion-strengthened materials based on nickel - 20-percent chromium (Ni-20Cr) invariably introduce additional amounts of oxygen. By being able to preselect close to optimum cleaning conditions, the time required for complete impurity removal by the reduction of oxides may be appreciably shortened.

Specifically, the data suggest that the Cr_2O_3 present in oxidized Ni-20Cr or mixed NiO-Cr_2O_3 can be reduced with dry hydrogen or by a combination of dry hydrogen and carbon additions to the powders. Furthermore, the results indicate that the reduction of Cr_2O_3 with either of these reducing conditions is facilitated by the presence of nickel. This information should be useful in the preparation of dispersion-strengthened Ni-20Cr and perhaps also the more complex nickel-base superalloys containing chromium.

SUMMARY OF RESULTS

This study, designed to determine the effect of controlled hydrogen, carbon, and hydrogen-carbon reductions of the oxides present in nickel - 20-percent chromium at temperatures to 1200° C, produced the following results:

1. Hydrogen, carbon, and hydrogen-carbon were all effective in reducing nickel (nickelous) oxide, but complete reduction at the lowest temperature was achieved with hydrogen alone.

2. While hydrogen plus carbon permitted the reduction of chromic sesquioxide to go near to completion, only about 30-percent reduction could be achieved with hydrogen or carbon alone.

3. Mixed nickel (nickelous) oxide - chromic sesquioxide in the ratio 80-percent nickel - 20-percent chromium is effectively reduced with either hydrogen or hydrogen-carbon. The reduction occurs at about the same rate and over the same temperature range. Carbon alone is not as effective in reducing the mixed oxides.

4. Oxidized nickel - 20-percent chromium is effectively reduced by hydrogen, carbon, and hydrogen-carbon. However, the reduction starts at a lower temperature and is more complete with either hydrogen or hydrogen-carbon than with carbon alone.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 10, 1972,
501-21.

REFERENCES

1. Weeton, John W.; and Quatinetz, Max: Cleaning and Stabilization of Dispersion Strengthened Materials. Oxide Dispersion Strengthening. George S. Ansell, Thomas D. Cooper, and Fritz V. Lenel, eds., Gordon and Breach Science Publ., 1968, p. 751.
2. Fedorchenko, I. M.: Factors Which Disturb Normal Shrinkage in the Sintering of Powdered Metals. Soviet Phys.-Tech. Phys., vol. 1, no. 9, Sept. 1957, pp. 2006-2113.
3. Grant, Nicholas J.; Siegel, Howard J.; and Hall, Robert W.: Oxide Dispersion Strengthened Alloys. NASA SP-143, 1967, p. 10.
4. Schafer, Robert J.; Quatinetz, Max; and Weeton, John W.: Some Effects of Variation of Hot-Pressing Temperature Upon the Agglomeration of MgO in a Dispersion Strengthened Nickel-MgO Alloy. NASA TN D-1167, 1962.
5. Strater, Kurt; and Mantell, Charles L.: The Kinetics of Hydrogen Reduction of Chromic Oxide. Trans. AIME, vol. 230, no. 5, Aug. 1964, pp. 1141-1150.
6. Darling, A. S.: Low Pressure Metallurgy. The Reduction of Oxides by Carbon. Metallurgia, vol. 64, no. 382, August 1961, pp. 71-75.
7. Herbell, Thomas P.: Hydrogen Halide Cleaning of Powder Metallurgy Nickel-20 Chromium-3 Thoria. Int. J. Powder Met., vol. 8, no. 1, Jan. 1972, pp. 29-41.

8. Herbell, Thomas P.: Thermogravimetry System Designed for Use in Dispersion Strengthening Studies. Thermochim. Acta, vol. 4, August, 1972, pp. 295-307.

9. Garn, Paul D.; Geith, Charles R.; and DeBala, Stephen: Furnace Mounting and Control System for Ainsworth Vacuum Automatic Recording Balance. Rev. Sci. Inst., vol. 33, no. 3, Mar. 1962, pp. 293-297.

10. Hurd, Dallas T.: An Introduction to the Chemistry of the Hydrides. John Wiley & Sons, Inc., 1952, p. 103.

11. Hopkins, W. D.: Physical Chemistry and Metal Extraction. J. Garnet Miller, London, 1954. (Referenced in Jones, William D.: Fundamental Principles of Powder Metallurgy. Edward Arnold, London, 1960, p. 546.)

12. Jones, William D.: Fundamental Principles of Powder Metallurgy. Edward Arnold, London, 1960, p. 547.

13. Meyer, Hans H.: The Reduction of Chromium Oxide and the Preparation of Carbon-Free Chromium-Iron Alloys. Mitt. Kaiser-Wilhelm-Inst. Eisenforsch. Düsseldorf, vol. 13, 1931, pp. 199-204. (Referenced in Strater, Kurt; and Mantell, Charles L.: The Kinetics of Hydrogen Reduction of Chromic Oxide. Trans. AIME, vol. 230, no. 5, Aug. 1964, p. 1142.)

14. Baukloh, W.; and Henke, G.: Reduction of Chromic Oxide with Hydrogen and Solid Carbon. Z. Anorg. Allgem. Chem., vol. 234, 1937, pp. 307-310. (Referenced in Strater, Kurt; and Mantell, Charles L.: The Kinetics of Hydrogen Reduction of Chromic Oxide. Trans. AIME, vol. 230, no. 5, Aug. 1964, p. 1142.)

15. Barshchevskaya, L. F.; and Radomysel'skii, I. D.: Process of Coreduction of Iron and Chromium Oxides with Hydrogen. Soviet Powder Met. Metal Ceramics, vol. 10, no. 2, Feb. 1971, pp. 85-90.

TABLE I. - WEIGHT LOSS AND RESIDUE DESCRIPTION FOR CONTROLLED HYDROGEN, CARBON, AND HYDROGEN-CARBON REDUCTIONS OF OXIDES IN NICKEL - 20-PERCENT CHROMIUM

Sample	Reducing agent		
	Hydrogen	Carbon	Hydrogen-carbon
	Percent of calculated weight loss for reduction of metal (Residue coloration)		
NiO	[a]100.0 (grey metallic)	[b]95.4 (black)	[a]98.2 (grey metallic)
Cr_2O_3	[c]30.1 (green)	[d]29.0 (black)	[e]97.2 (grey)
NiO + Cr_2O_3 (80Ni-20Cr)	[f]97.8 (silver metallic)	[g]34.0 (green)	[f]102.3 (silver metallic)
NiO + $NiCr_2O_4$ (oxidized Ni-20Cr)	[f]100.0 (silver metallic)	[h]72.0 (black)	[f]103.0 (silver metallic)

Footnotes denote X-ray diffraction analysis of reduction residue:

[a] Ni.

[b] Ni, NiO.

[c] Cr_2O_3, Cr.

[d] Cr_2O_3.

[e] Cr.

[f] Ni solid solution (20Cr).

[g] NiO, $NiCr_2O_4$, Ni solid solution (slightly less than 20Cr).

[h] Ni solid solution (low chromium), Cr_2O_3.

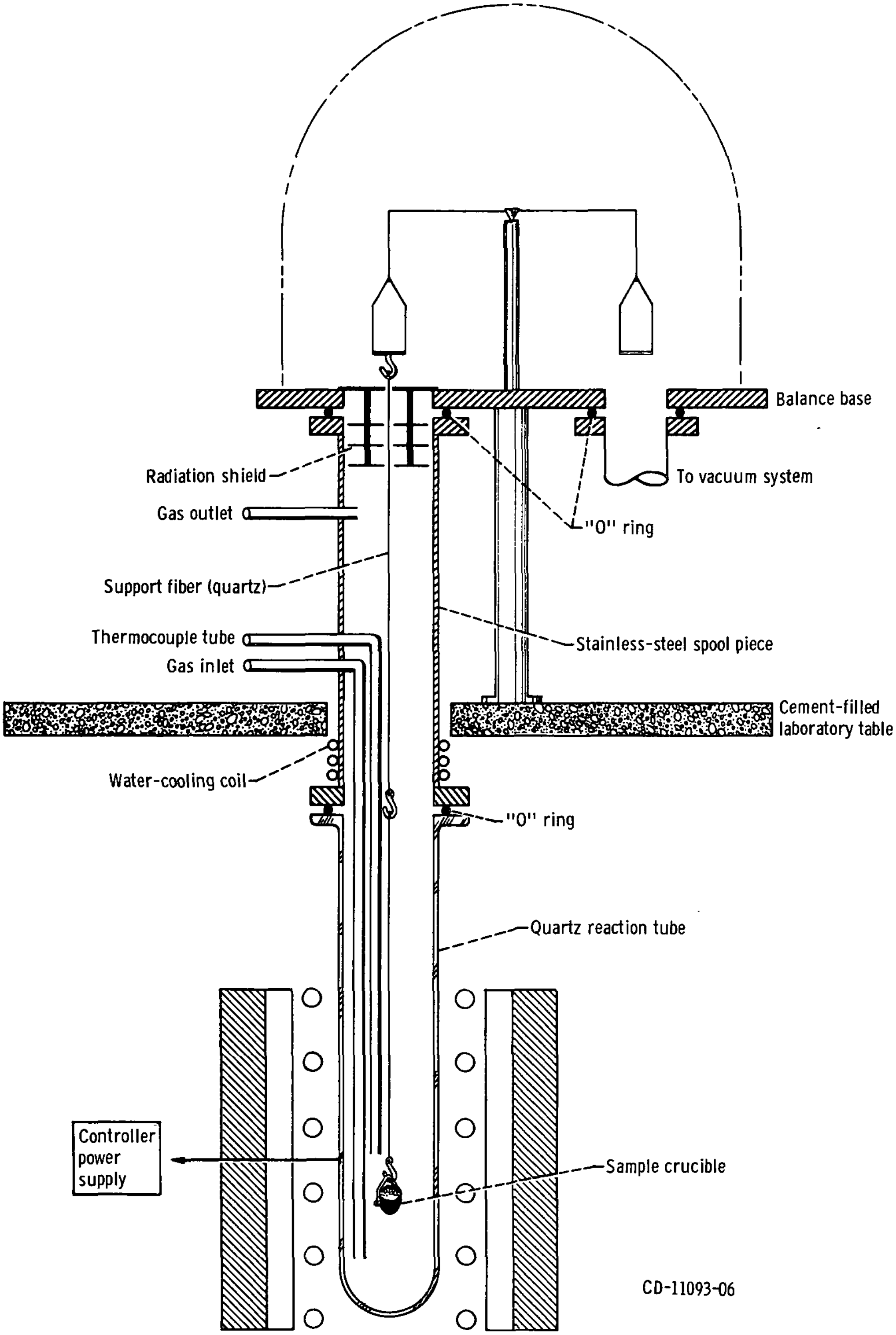

Figure 1. - Reaction tube assembly.

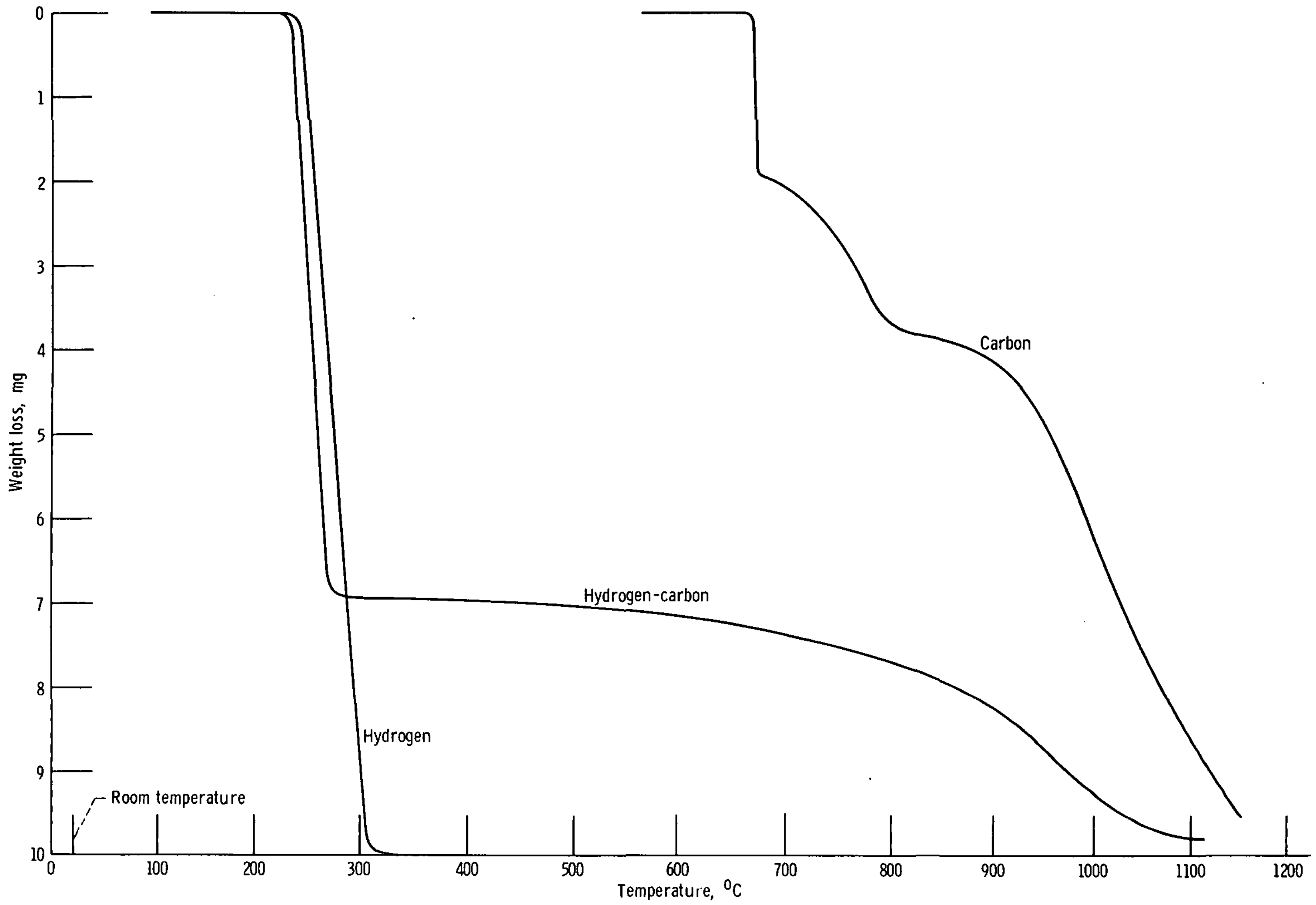

Figure 2. - Controlled reaction of hydrogen, carbon, and hydrogen-carbon with nickel (nickelous) oxide.

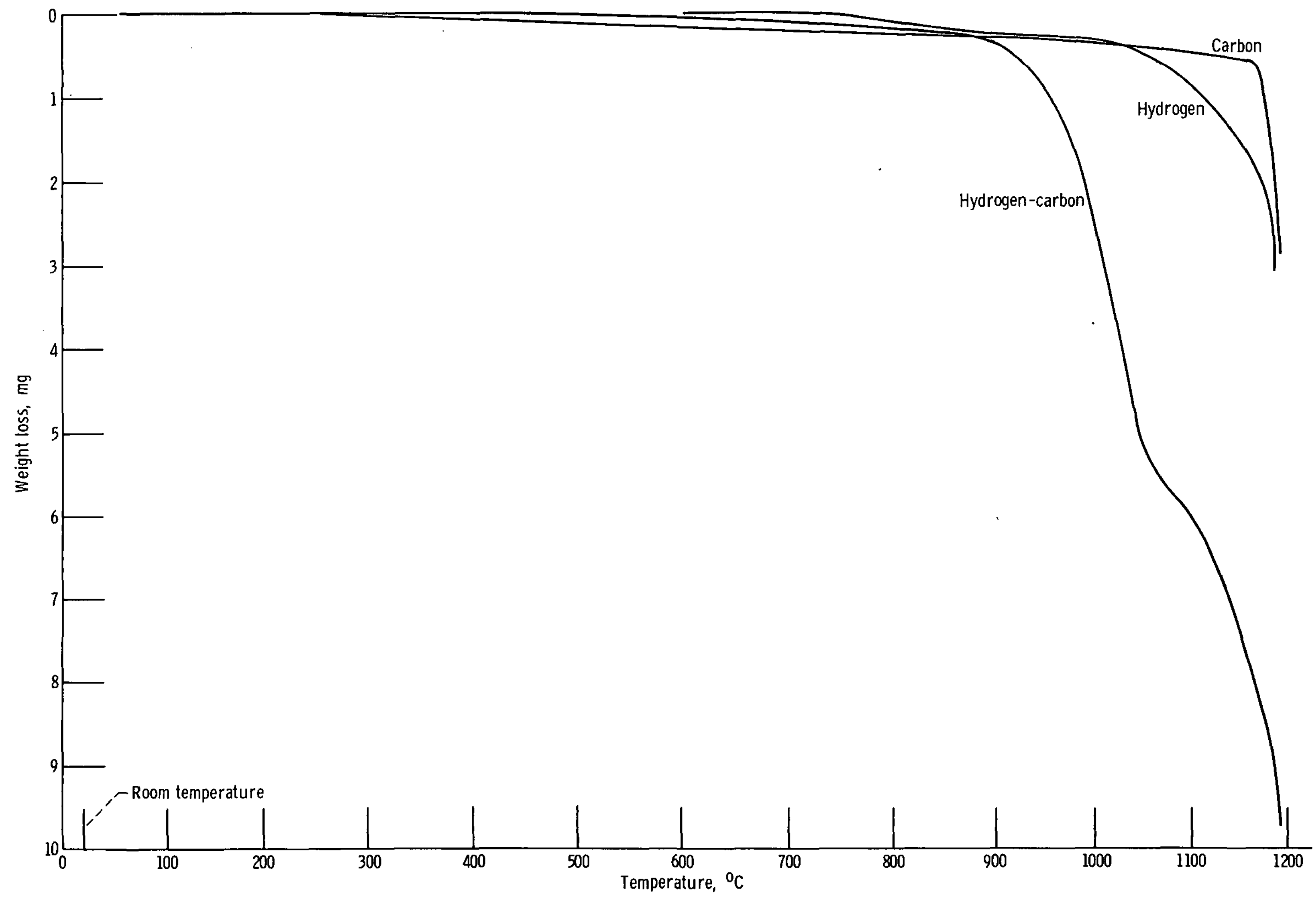

Figure 3. - Controlled reaction of hydrogen, carbon, and hydrogen-carbon with chromic sesquioxide.

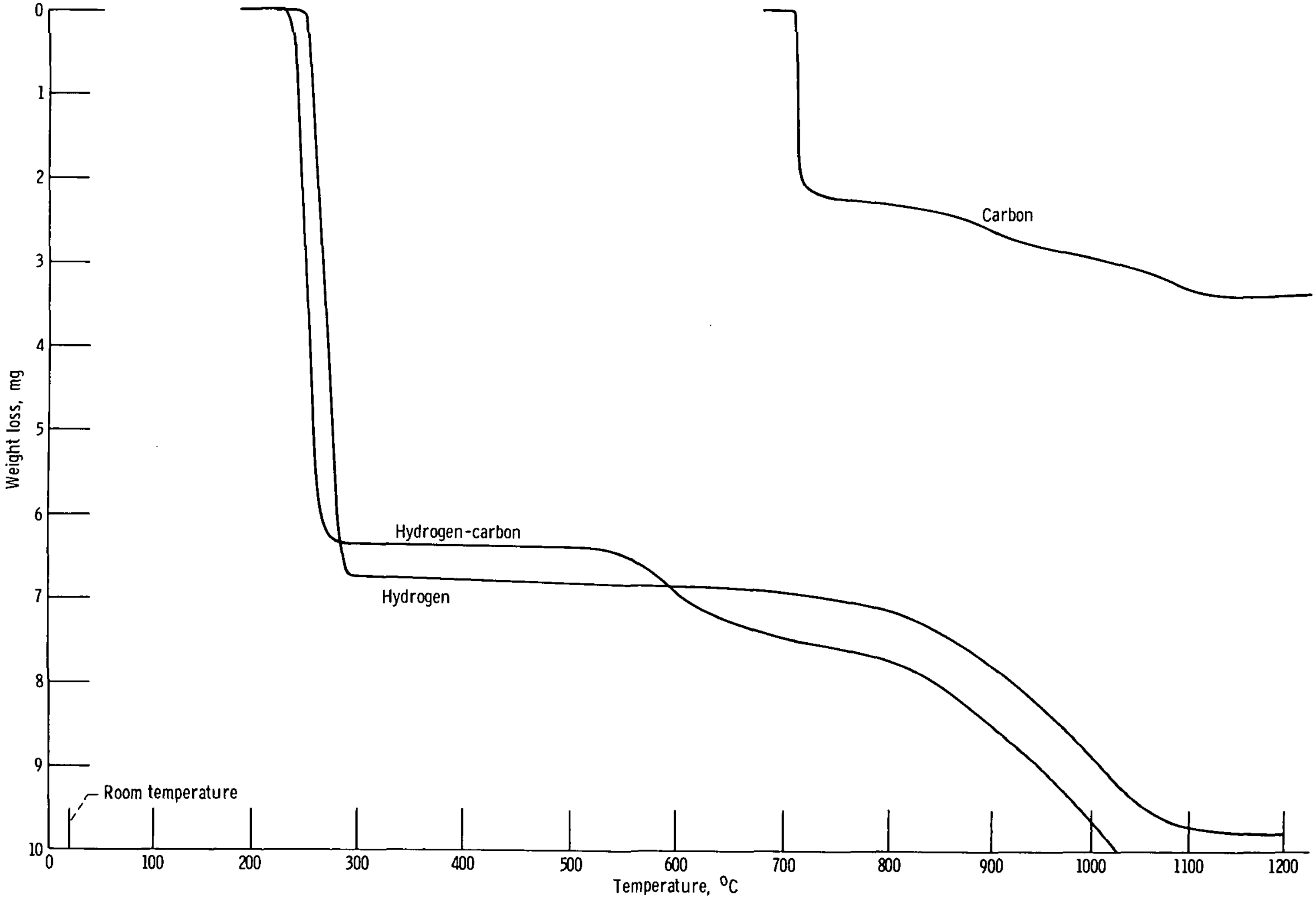

Figure 4. - Controlled reaction of hydrogen, carbon, and hydrogen-carbon with mixed nickel (nickelous) oxide and chromic sesquioxide (80Ni-20Cr).

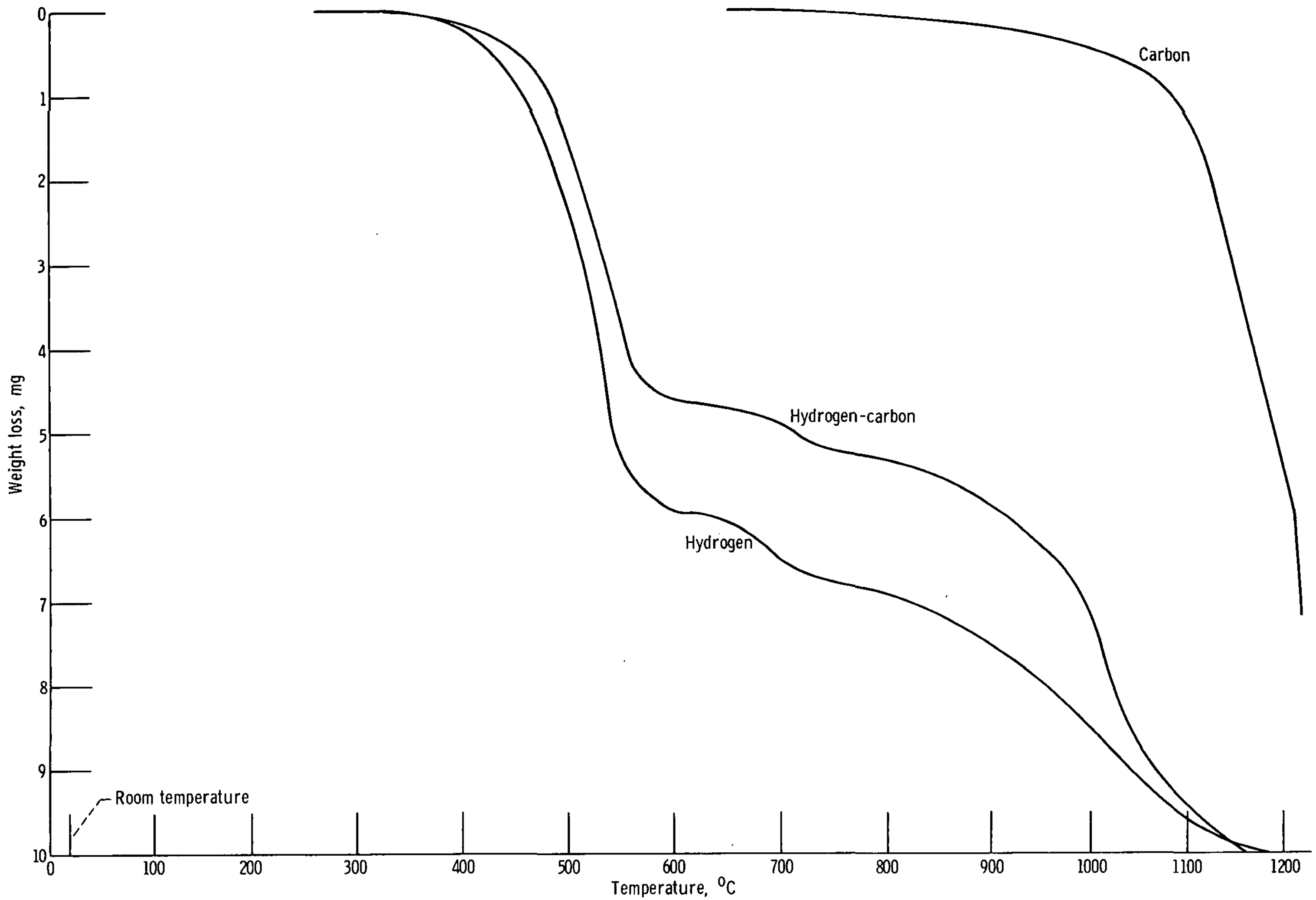

Figure 5. - Controlled reaction of hydrogen, carbon, and hydrogen-carbon with oxidized nickel - 20-percent chromium.

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