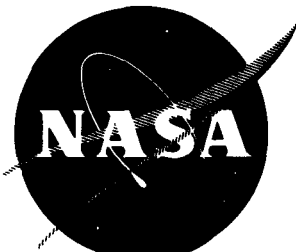


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DISPERSION CHROMIUM PRODUCT

by

M. L. Gimpl and N. Fuschillo

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-7608

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Summary Report

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March 1967

CONTRACT NAS 3-7608

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DISPERSION CHROMIUM PRODUCT

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ABSTRACT

Two methods of producing dispersion chromium alloy product were investigated. First, magnesia (MgO) was coated with chromium by chemical vapor deposition from dicumene chromium in a fluidized bed. This method produced high carbon chromium product. The carbon content varied from 0.3 percent to as high as 6.0 weight percent. Second, MgO particles were coated with chromium chloride hexahydrate ($[\text{Cr}(\text{H}_2\text{O})_6]\text{Cl}_3$) by evaporating to dryness a suspension of the MgO particles in solution with chromium chloride hexahydrate. The water of hydration was driven off under a chlorine-rich atmosphere. The composite of chromium chloride (CrCl_3) and MgO was then reduced under a dry hydrogen atmosphere. Heating of a pressed sample of the composite at 2400°F (1316°C) caused the MgO particles to combine with the chromium oxide present to form a spinel.

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DISPERSION CHROMIUM PRODUCT

by

M. L. Gimpl and N. Fuschillo

Section 1

SUMMARY

The main emphasis in this program was on the production of dispersion-hardened chromium by chemical vapor deposition in a fluidized bed. Fine particles (< 0.1 micron in diameter) of magnesia (MgO) or thoria (ThO_2) were fluidized with argon and/or hydrogen in a heated column. Dicumene chromium ($Cr(C_9H_{12})_2$) in the vapor state was passed into the column and decomposed. This caused plating of the individual oxide particles. After plating was complete, the individually coated oxide particles were sintered together to form a composite particle approximately 50 microns in diameter. These large composite particles could be safely handled in air without oxidation. The vapor plating system is shown in figure 1.

Microscopic analysis showed the method produced a very uniform dispersion of particles. Chemical analysis showed that the lowest carbon content of the dispersion-chromium coatings obtainable in this study was 0.3 wt. percent. After heat treating the deposit in a vacuum at $1000^\circ C$ for 1 hour, an X-ray diffraction analysis showed $Cr_{23}C_6$ and MgO lines. The prognosis for producing low-carbon-content chromium coatings by vapor plating with dicumene chromium is poor. The use of chromium iodide (CrI_3) as a precursor compound produced coatings at a rate too slow to be considered practical for the small system.

Dispersion chromium product was also produced by precipitating chromium chloride hexahydrate, ($[Cr(H_2O)_6]Cl_3$) onto fine-particle magnesia in an aqueous solution. The water of hydration was removed by heating in a chlorine atmosphere. The magnesia — anhydrous-chromium-chloride ($MgO-CrCl_3$) mixture was then reduced in dry hydrogen at $1020^\circ C$.

Microscopic analysis showed a homogeneous distribution of magnesia particles in a chromium matrix product. Heating at $2400^\circ F$ ($1316^\circ F$) for 10 hours in vacuum caused the MgO particles to react with the chromic oxide (Cr_2O_3) present to produce a spinel phase. The MgO particles could not be detected by electron microscopy after the heat treatment.

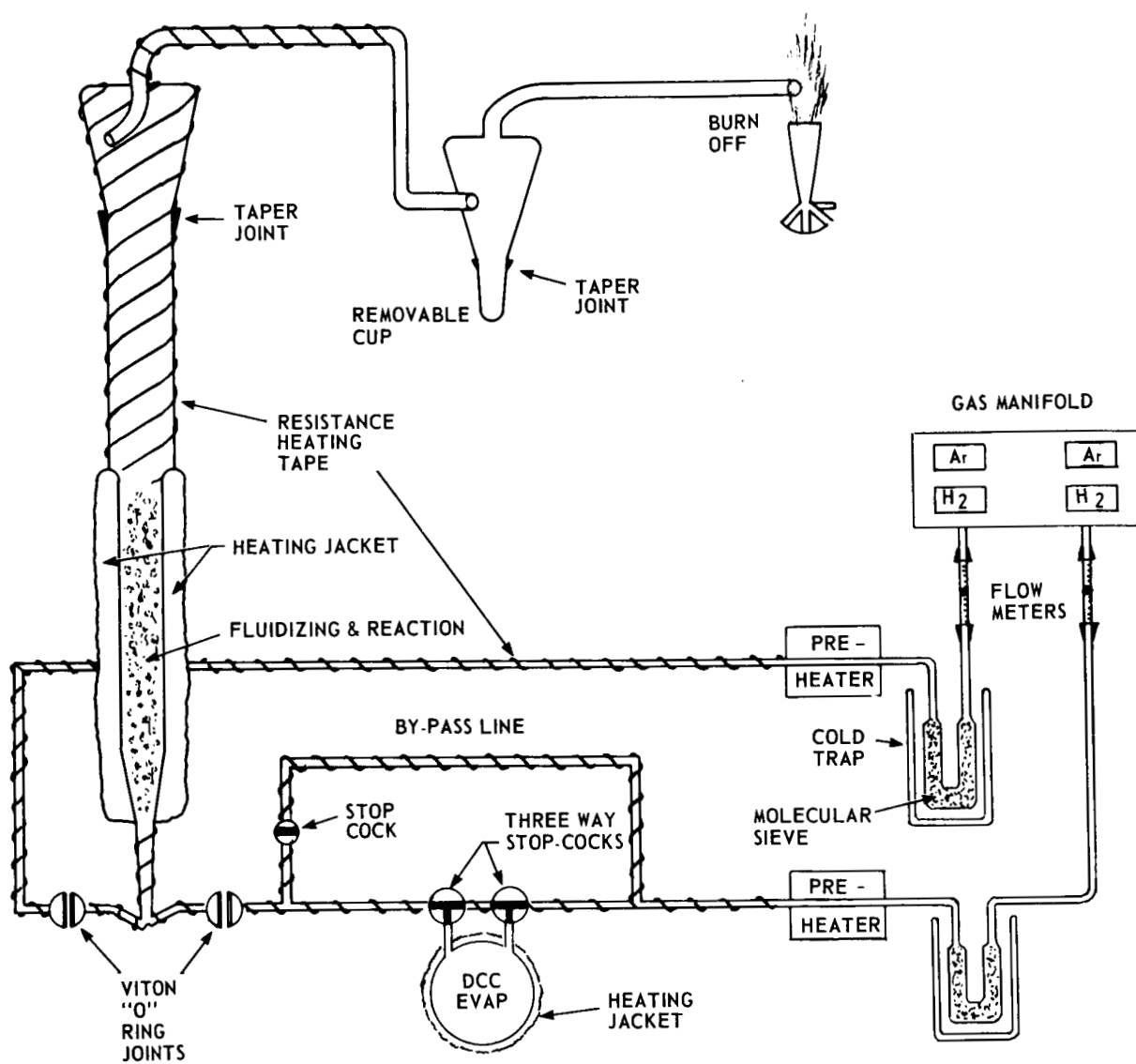


Figure 1. Apparatus Used to Produce Dispersion Chromium Product in a Fluidized Bed

Section 2

INTRODUCTION

In recent years, chromium has been considered as a possible base metal for alloy systems for the use in the 2000° to 2500°F range. Among the desirable properties that qualify chromium for such a task are its high melting point (3404°F), high elastic modulus (41×10^6 psi), relatively low density (7.2 gm/cm^3), good thermal conductivity, low coefficient of thermal expansion, and good oxidation resistance. However, chromium, like several other body-centered cubic metals, suffers from a relatively high transition temperature for ductile-to-brittle fracture which has hindered its use in engineering applications.

Chromium can be strengthened by solid-solution alloying with a resultant increase in transition temperature. It can also be strengthened by dispersions of a second phase obtained by precipitation from solid solution (precipitation hardening) or by an inert particle distribution (SAP-type strengthening.) Chromium-magnesia dispersions have been made and are reported to result in strengthening, while concurrently reducing the transition temperature.¹

The principal disadvantage of these chromium-magnesia composites is the coarse particle size of magnesia, which tends to minimize the dispersion-strengthening effect. While it has been found that micron-size particles can give some strengthening, little doubt remains that particles in the 0.01- to 0.1-micron range are the most effective for the high temperature strengthening needed in long-time applications under creep conditions. It remains to be seen what effect a decrease in magnesia particle size will have on the transition temperature of the composite, and whether ductility near room temperature can be maintained.

It was therefore the goal of this program to produce dispersions of less than 8 vol percent of submicron particle MgO and ThO₂ in chromium. The methods used were vapor plating in a fluidized bed and reduction of a chromium salt precipitated onto submicron-particle (< 0.1 micron) MgO.

The materials produced were evaluated and found not to meet the metallographic and chemical requirements of the program.

¹C. G. Reed and W. L. Schallil, "Chromium-Magnesia Composites," J. of Metals 16, 175 (1964).

Section 3

METHODS

3.1 Chemical Vapor Deposition in a Fluidized Bed

The method used in this program for the preparation of dispersion-hardened alloys was essentially a chemical-vapor deposition process. In this process, a volatile compound of chromium, dicumene chromium, was vaporized, passed into a chamber containing a fluidized bed of the dispersoid material, and decomposed. The chromium resulting from the decomposition was deposited on the dispersoid particles to form the alloy powder, which was then sintered and evaluated by chemical, analytical and metallographic methods.

The apparatus used in this process is shown schematically in figure 1. This apparatus was evolved from a series of experiments to improve the design of the fluidized bed. The design of the fluidized bed is discussed in the appendix. In the column are placed 7 to 10 grams of dispersoid (MgO to ThO_2) and fluidized with a stream of a mixture of argon and hydrogen. A flow of approximately 2 liters/min is necessary for fluidization of the bed (2-inch diameter) illustrated in figure 1.

Pure hydrogen and pure argon obtained from gas cylinders are passed through molecular sieve traps held at dry-ice temperature (to remove traces of moisture), heated, and then passed through flow meters. Two separate streams of hydrogen and two separate streams of argon are used. The four streams of gas are then blended into two streams, each stream containing a mixture of ratio quantities of hydrogen and argon. One stream becomes saturated with dicumene chromium as the gas is passed through the vaporizer pot and heated to 180° to 200°C . Hydrogen iodide (HI) may be bled into the other stream of gas. The two streams of gas are then blended together at the "T" and passed into the reactor column, where the dicumene chromium is decomposed and the chromium is plated onto the oxide particles. At the end of the plating period, the gas flow rate is lowered (to about 1 to 1.5 liters/min) to partially collapse the bed. The reactor temperature is then increased to 375° to 400°C and held there for 1/2 hour to sinter the plated particles so that the surface-to-volume ratio is reduced. This reduces oxidation to a minimum when the chromium-coated particles are exposed to air. The sintered particles are approximately 50 to 100 microns in diameter; the unsintered fine particles are pyrophoric.

Hydrogen is used as a carrier gas to reduce the decomposition temperature. Hydrogen alone does not properly fluidize the column. Therefore, a mixture of 50 percent H_2 — 50 percent Ar is used to keep the reduction temperature low and fluidize the column.

The use of the "T" at the bottom of the column in figure 1 is essential for blending gases and proper fluidization. A catalyst such as HI is added to the dicumene chromium stream in the "T" to catalyze the deposition process. Addition before this point causes decomposition of the dicumene chromium in the line rather than in the column.

This process produced uniform dispersions; however, the use of dicumene chromium has certain disadvantages that outweigh its advantages. The main advantage is the volatility of dicumene chromium at low temperatures (below $200^{\circ}C$) and its ease of decomposition at temperatures below $350^{\circ}C$. Since decomposition starts at about $210^{\circ}C$, this temperature was the upper temperature limit of the vaporizer pot and dicumene chromium lines.

Among the disadvantages of dicumene chromium are a low chromium content (18 wt percent chromium) and the deposition of carbon in the chromium coatings. The tendency to decompose readily becomes a problem if the vaporizer and line become too hot (above $200^{\circ}C$). The dicumene chromium then decomposes to a chromium sludge, which blocks the lines, and cumene (isopropyl benzene), which is quite volatile (b.p. $150^{\circ}C$) and becomes a source of carbon contamination.

The codeposition of carbon in the chromium coatings is the main disadvantage of dicumene chromium. Up to 8 wt percent of carbon can be obtained in the coatings using a straight thermal decomposition process in an argon atmosphere. The use of hydrogen gas enables the decomposition temperature to be lowered from 350° to about $300^{\circ}C$, thus alleviating the carbon problem somewhat. When this is done, the carbon content of the coatings still ranges from 3 to 4 percent. Further carbon reduction is by the use of an HI catalyst. The action of the catalyst is twofold: (1) the process temperature is reduced to $250^{\circ}C$, thereby reducing thermal cracking of the hydrocarbon, and (2) HI reacts with deposited carbon forming a volatile iodide. Using both hydrogen gas and hydrogen iodide catalyst with a HI—to—dicumene-chromium ratio of 1:4, the carbon in the coating is reduced to 0.3 wt percent; some of the chromium, however, is converted to CrI_3 by this process.

System problems were also encountered. The chromium is deposited at the extremely low rate of 1.5 grams per hour, owing to the inability of bringing dicumene chromium into the reactor at a faster rate. If a fast gas flow is used, the submicron-sized dispersoid powders blow out of the reactor. Even at a flow rate of 2 liters/min, there is considerable blowover. This may be minimized by expanding the column volume as the column height increases and by providing a large expansion chamber at the top of the column. Another factor which limits the amount of dicumene chromium

flowing into the column is the vaporizer temperature. For reasons previously stated, the upper limit of this temperature is 200°C. Thus, with a flow rate of 1 liter/min and a vapor pressure of 10 mm Hg, the plating rate is limited to 1.5 grams of chromium per hour. By increasing the vaporizer temperature and flow rates slightly, plating rates of 2 to 2-1/2 grams per hour are obtained. This was found to be the maximum practical rate. Because of this, long plating times are necessary to obtain appreciable amounts of plated powder. Approximately 30 grams of powder can be obtained in a 30-hour run.

It takes approximately 3 hours from the start of a run until the optimum vaporization temperatures and plating temperatures are attained. About 3 hours are required for shutdown; this involves cooling the vaporizer, flushing hydrogen out of the system, sintering the powder, and cooling the column. During a run, it is necessary to bypass the vaporizer pot for about 5 minutes every hour to flush condensed dicumene chromium out of the lines. In a 30-hour run, starting with 7 grams of dispersoid material, 30 grams of product containing about 25 vol percent of dispersoid material are obtained.

Although the slow plating rate is the major limitation on this process, the blowover of powder from the column is an important consideration. Since the amount of blowover cannot be controlled or predicted, the composition of the final product cannot be determined in advance. The fine oxide particles and plated particles become entrained in the gas stream and act very much like an aerosol. To minimize this, a series of volume expansions of the column was used to lower the linear flow rate of the gas. The column was continually vibrated to cause materials adhering to the column walls to be shaken loose and dropped back into the bed. Even with these precautions, a flow rate of 2 liters/min still caused a 50-percent blowover in a 30-hour period. The material that blows over cannot be recovered since it is wet with cumene, a major source of carbon contamination.

The fluidized bed process requires constant supervision of the gas flows and temperatures. Any decrease in the gas flow rates, however brief, may cause the bed to collapse. The temperatures must be closely controlled to prevent decomposition or condensation of dicumene chromium in the lines or plating of chromium on the reactor walls. The surface area of the powder is much greater than the wall surface area at the beginning of a run; however, if too much blowover occurs, the reverse becomes true and plating will occur on the walls. If the reactor is too hot, plating takes place on the reactor wall at the bottom of the bed where the reactor column is relatively narrow. Plating on the column walls cannot be tolerated; the particles in the column tend to adhere to the plated walls, reducing the ratio of particles surface area to wall surface area, which further tends to increase the rate of wall plating.

Because of these disadvantages, it is not practical to produce laboratory quantities (1 to 2 lb) of dispersion-hardened material by the fluidized bed process. At present it takes approximately 100 hours of continuous operation to produce 0.5 lb of material in a 2-inch-diameter bed. A 6-inch-diameter bed would permit a flow rate of approximately 8 times that of the 2-inch bed; thus, in 100 hours, approximately 4 lb of material should be produced.

3.2 Results of Fluidized Bed Operations

Ten runs were made in reactors similar to that shown in figure 1. The parameters used in the operation of the reactor in each of the runs are listed in table 1. Several other runs were made which did not yield a product which could be analyzed.

The temperatures shown in table 1 are average temperatures that were controlled with a Variac potentiometer. Temperature variations of $\pm 5^{\circ}\text{C}$ from the mean over a 20-hour run were common. At the end of each run, the reactor temperature was raised approximately 50°C to permit sintering of the particles before removal.

3.3 Chemical Precipitation and Reduction

An alternative technique to the fluidized-bed technique of producing dispersion chromium product is the precipitation of a chromium salt around MgO particles followed by selective reduction.

The following procedure was used in preparing the dispersion chromium product. Two grams of 0.1-micron MgO were suspended in water. The suspension was heated and 477 grams of $[\text{Cr}(\text{H}_2\text{O})_6]\text{Cl}_3$ were dissolved in the water-MgO suspension. The mixture was transferred to a container, stirred, and kept under a chlorine atmosphere. The stirred mixture was heated to 230°C to drive off the excess water. The temperature was then raised to 550°C for 15 hours to remove the water of hydration producing anhydrous CrCl_3 with MgO. Water removal is done under a chlorine atmosphere to prevent the $[\text{Cr}(\text{H}_2\text{O})_6]\text{Cl}_3$ from converting to Cr_2O_3 .

The MgO- CrCl_3 mixture was reduced in dry hydrogen at 1020°C for 4 hours. This treatment produced a dispersion chromium product without any trace of Cr_2O_3 . Improperly dried hydrogen caused some batches to contain an excess of Cr_2O_3 .

To insure dry hydrogen, Olin Mathieson prepurified grade (dew point, -95°C) was used. The hydrogen is passed through a deoxo unit and then into a dry ice—chloroform—carbon-tetrachloride cold trap containing a molecular sieve before entering the system.

TABLE 1. PARAMETERS USED ON RUNS IN WHICH
DISPERSION PRODUCT WAS COLLECTED

Run #	Temperatures (°C)			Argon or 50-50 H ₂ -Argon flow rate (liter/hr)*	HI flow rate (liter/hr)	Remarks
	DCC evaporator	Bottom "T" of column	Column			
1	180	200-297	405	2.4	--	Excessive blow- over and Cr plate on reactor walls
2	200	280	395	2.5	--	10.6 g of product
3	210	195	350	2.6	--	9.3 g of product
4	200	155	310	2.5	0.1	HI had moisture; run not completed (no product)
5	200	180	300	3.0	0.1	27 g collected
6	200	210	300	3.0	0.1	11 g collected; high iodine content
7	200	200	285	3.0	0.1	15 g collected
8	200	200	280	2.5	0.1	10 g collected
9	190	200	280	3.0	--	10 g collected
10	190	200	285	3.0	0.1	12 g collected

* The flow rates of each side of "T" joint were equal; this column is total flow rate.

Section 4

EVALUATION

4.1 Chemistry

Carbon analysis of the dispersion chromium product was done by the standard combustion technique.

Analyses of various runs are reported below in table 2 as percent of carbon present in the chromium product. It was not possible to reduce the carbon content below 0.3 wt percent with the HI catalyst, while without the use of the HI catalyst, the weight percent of carbon was never below 3.

Chromium content of the alloys was determined by standard wet chemical techniques.

X-ray analysis of as-deposited powder containing 4 wt percent C yielded only very broad peaks characteristic of an amorphous material and MgO peaks. Heat treating a sample in vacuum at 1000°C for 1 hour produced crystallization of the amorphous deposit. X-ray diffraction at this point produced Cr_{23}C_6 and MgO lines. No chromium lines were present.

Samples from runs using HI as a catalyst usually had significant iodine contents (>7 wt percent).

A sample of dispersion chromium product produced by the precipitation-reduction process contained 0.21 wt percent carbon. No other analyses were made. The material contained 3.8 volume percent MgO as determined by stoichiometry.

4.2 Metallography

4.2.1 Material Produced by Fluidized Bed Process

The sintered powder samples obtained from the fluidized-bed process were examined for particle size (figure 2) by placing a sample on a glass slide. The particles are from 20 to 100 microns in diameter (Run 2).

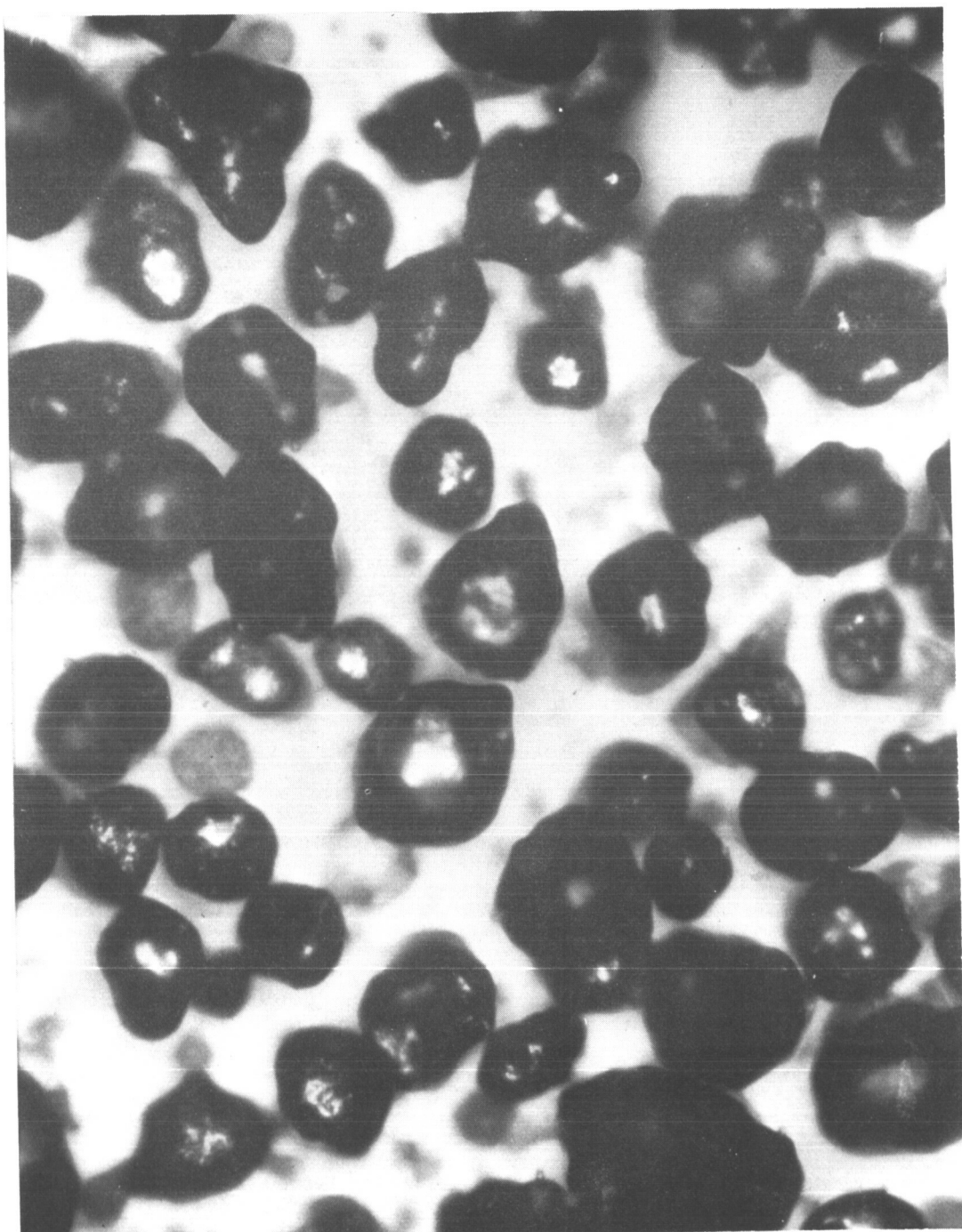


Figure 2. - Composite dispersion chromium product as obtained from the fluidized bed after sintering treatment (250X).

TABLE 2
ANALYTICAL CHEMISTRY OF VARIOUS RUNS

Run No.	Catalyst	Carrier gas	Carbon in Coating (Wt percent)	Wt. Percent Cr
1	None	Ar	6.0	NA
2	None	Ar	5.0	NA
3	None	50 Ar - 50 H ₂	3.0	NA
4	HI	50 Ar - 50 H ₂	NA	NA
5	HI	50 Ar - 50 H ₂	3.0	63
6	HI	50 Ar - 50 H ₂	2.4	6
7	HI	50 Ar - 50 H ₂	3	60
8	HI	50 Ar - 50 H ₂	NA	NA
9	HI	50 Ar - 50 H ₂	NA	NA
10	HI	50 Ar - 50 H ₂	0.3	21

For examination of the microstructure the powders were vacuum impregnated with epoxy. The impregnated samples were then ground on silicon carbide papers, polished with alumina, and finished with vibratory polishing with Linde B abrasive.

The MgO-containing dispersions were preferentially etched in hot phosphoric acid for 3 seconds. Cellulose acetate replicas were then made, which were shadowed with platinum and backed with carbon. After the cellulose acetate was dissolved, the replicas were viewed in the electron microscope.

Figure 3 shows a cross section of one of the composite particles as viewed in a light microscope (Run 2). The individual MgO particles are not quite capable of being resolved in the light microscope. Figure 4 shows a sintered particle observed under the electron microscope (Run 2). The individual particles are each coated with a metal product.

Figure 5 shows a sintered MgO composite particle after the temperature of the bed was raised to the sintering temperature before the chromium deposition was complete, causing the sintering of individual particles (Run 3). The remaining chromium was then deposited on the periphery of the composite.

4.2.2 Material Produced by Precipitation-Reduction Technique

Figure 6 shows a sample of Cr-3.8-vol-percent-MgO as prepared by the precipitation-reduction technique. The sample was reduced in dry hydrogen at 1030°C for 4 hours. This caused the particles of MgO to grow to ~0.2 micron diameters. Originally these particles were 0.1 micron in diameter.

Figure 7 shows a sample of Cr-3.8-volume-percent-MgO that has been pressed and sintered after the reduction treatment. The sample was pressed at 100,000 psi and sintered in a vacuum of 5×10^{-5} torr for 10 hours at 1315°C. Some Cr_2O_3 formed on the sample during the sintering operation. This was evidenced by the green coloring of the sintered compacts. The Cr_2O_3 has evidently reacted with the MgO particles. Electron microscopy showed no evidence of any remaining MgO particles.

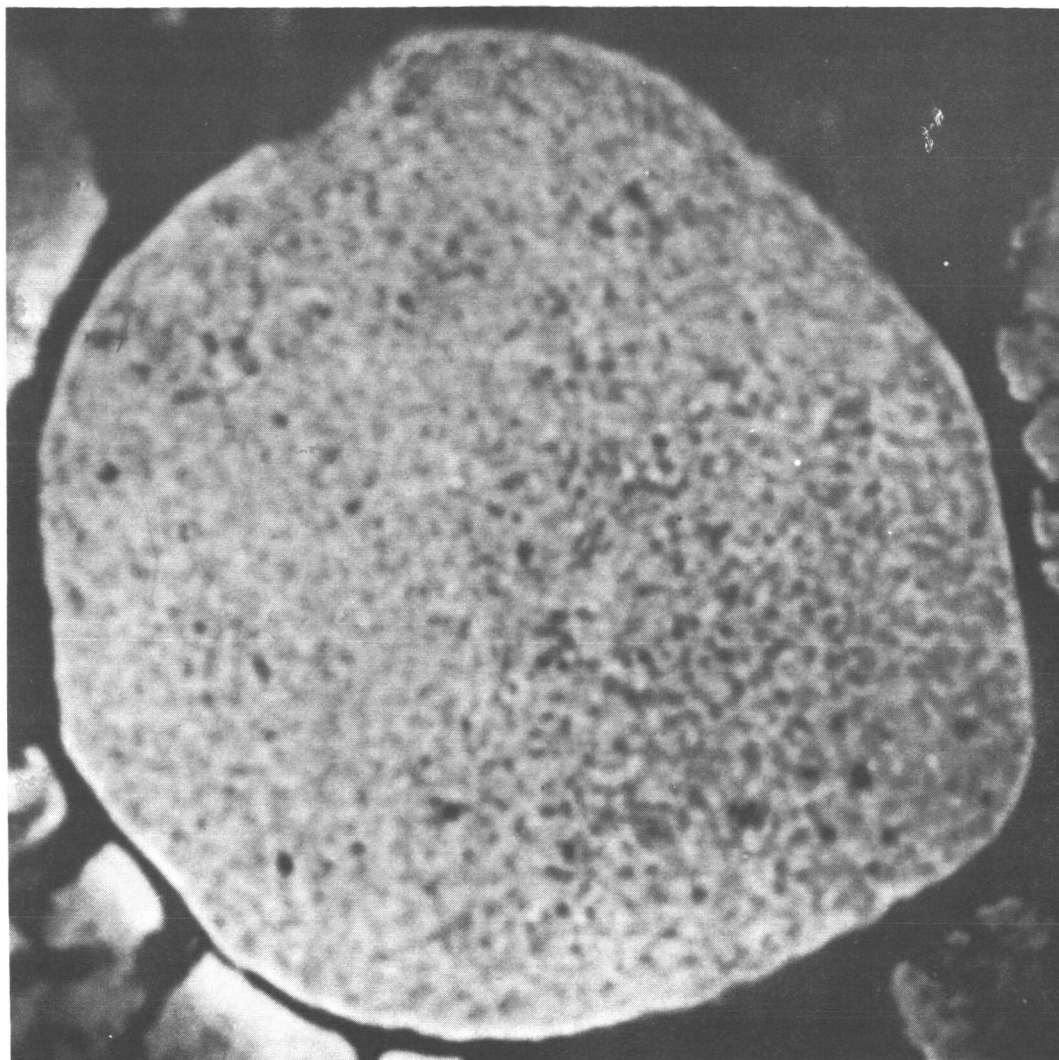


Figure 3. - Cross section of dispersion chromium product particle (2000X).

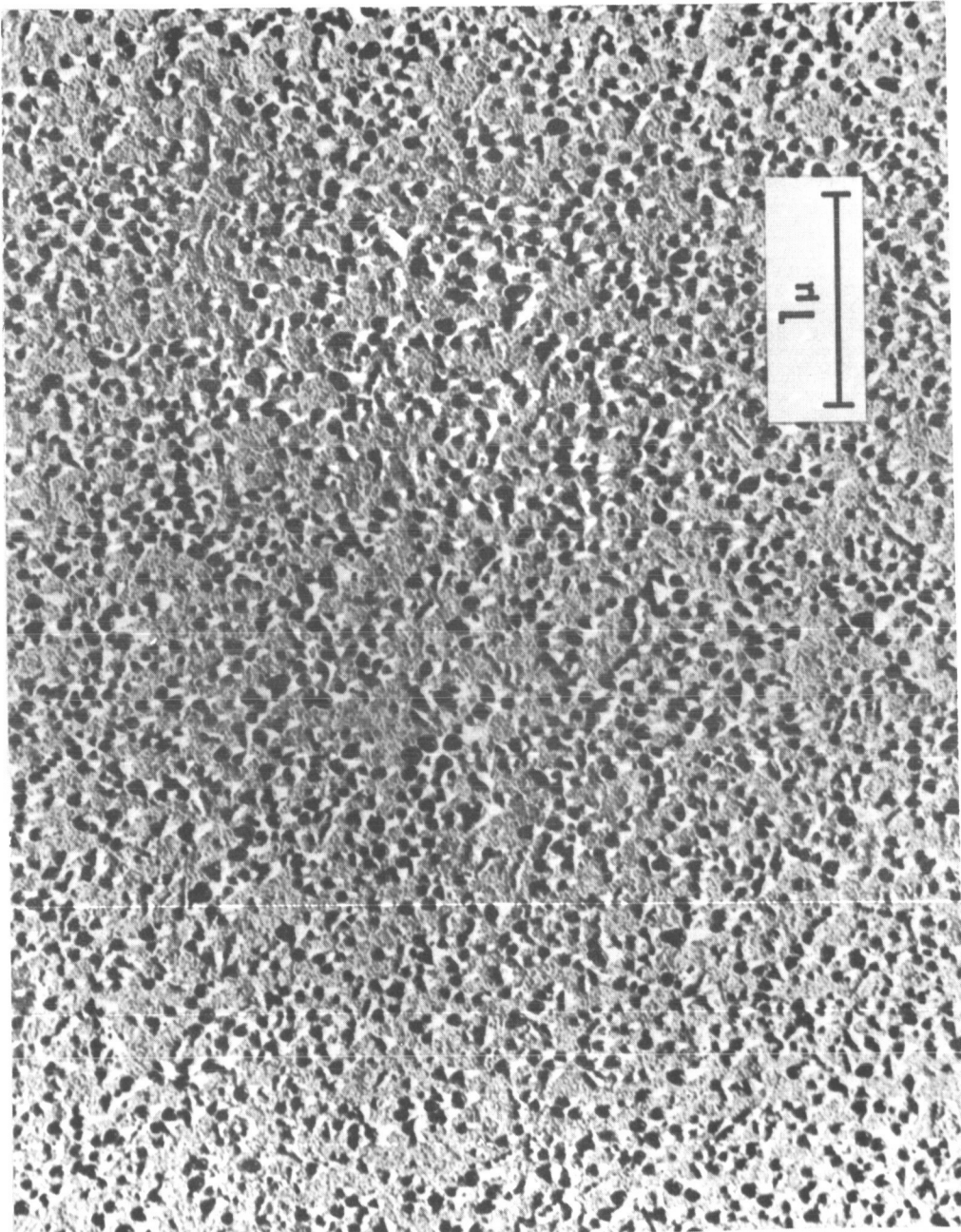


Figure 4. - Electron micrograph of dispersion chromium product (18,000X).

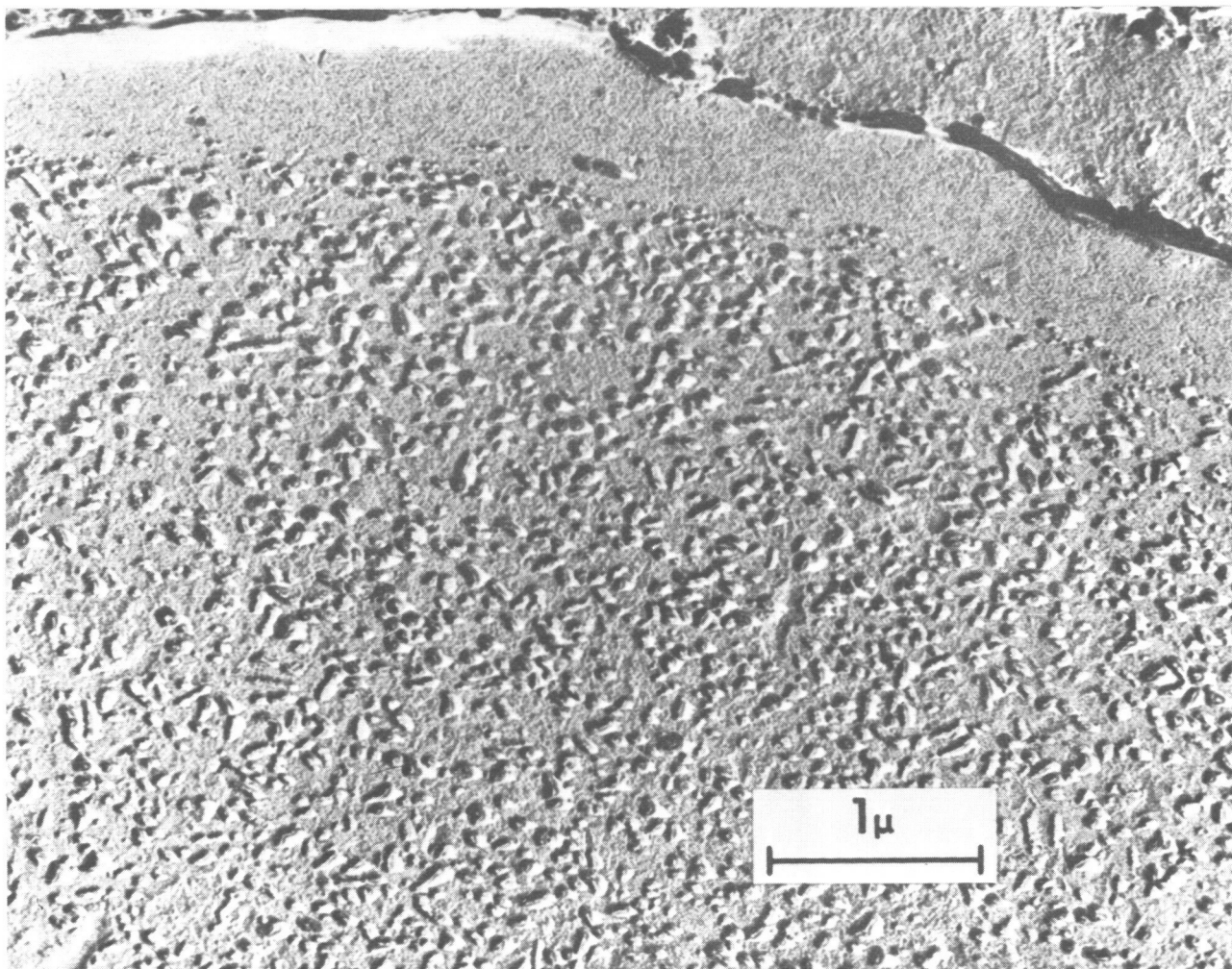


Figure 5. - Cross section of dispersion chromium particle, displaying premature sintering and subsequent chromium deposition of the periphery (18,000X).



Figure 6. - Electron Micrograph of CR-3.8-Volume-percent-MgO after hydrogen reduction (20,000X).

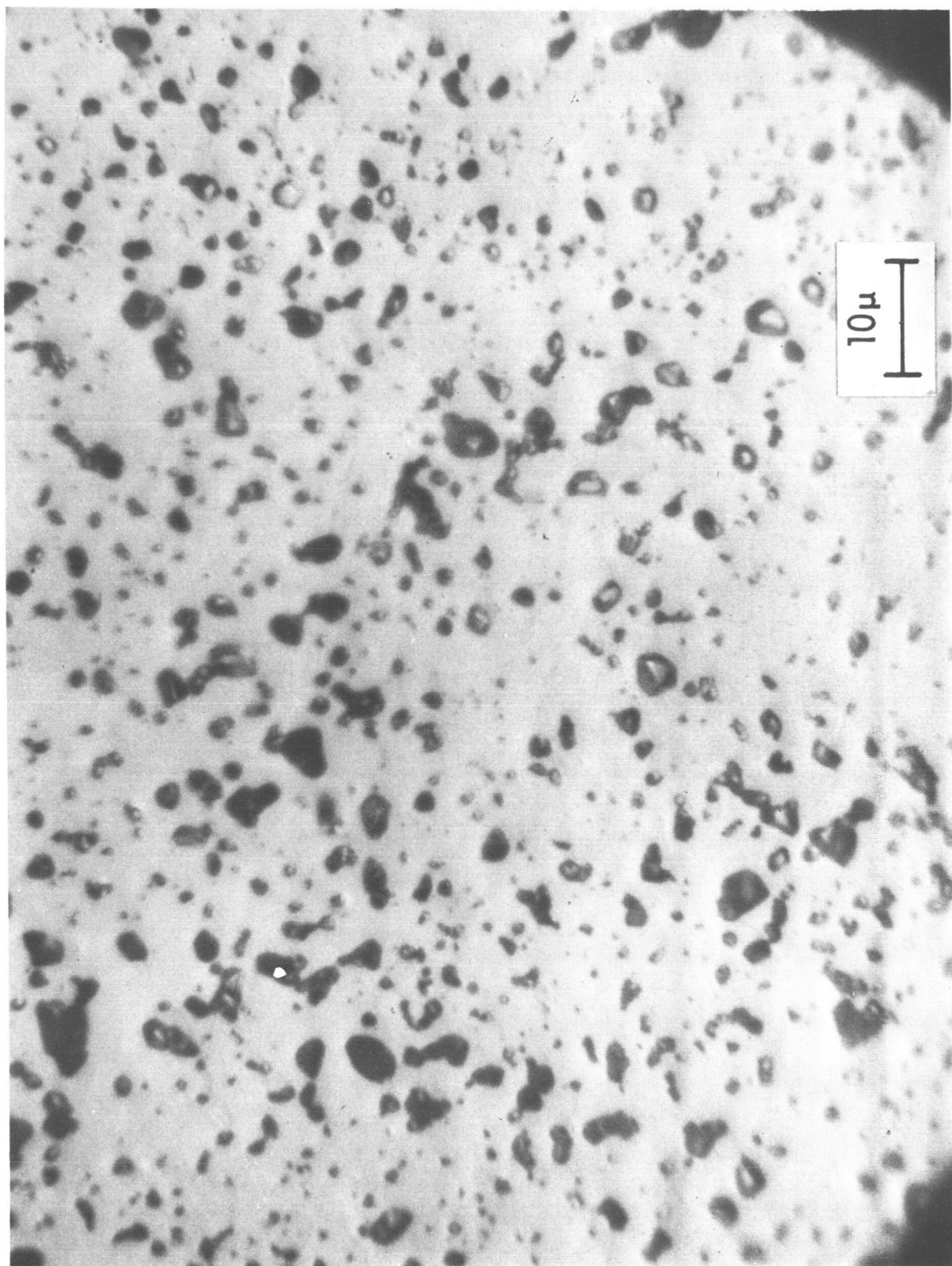


Figure 7. - Photomicrograph of Cr-3.8-volume-percent-MgO heated to 1315° C for 10 hours in vacuum (1000X).

Section 5

CONCLUSIONS AND RECOMMENDATIONS

Chromium chemical vapor deposition on fine MgO in a fluidized bed has been shown to produce a product with uniform particles in it.

The process has proved to be impractical in two respects: (1) it was not possible to produce coatings that contained less than 0.3 wt percent of carbon, and (2) the size of the column (2-inch-diameter) required long running times (more than 100 hours) to produce 0.5 pound of product.

The precipitation-reduction technique is capable of producing large amounts of low-carbon-content dispersion chromium product. The product produced in this manner has a good oxide particle homogeneity; the reduction step at 1030°C for 4 hours, however, causes MgO particles to grow from a 0.1-micron to a 0.2-micron diameter. The MgO particles will react with any Cr_2O_3 present to form a spinel.

It is recommended that future work with the latter process concentrate on alloy systems containing more stable particles such as the high-free-energy-of-formation carbides such as TaC and TiC, and borides such as B_4C and TiB_2 . A development effort would be necessary to make these borides and carbides available in sizes less than 0.1 micron in diameter and with the required chemical purity.

APPENDIX A
REACTOR DESIGN

Appendix A

REACTOR DESIGN

Several reactor designs were tried before the design described in section 3.1 was developed. The most critical part of the design is the lower part where the gas stream enters to support the bed. Some of the various designs tried are shown in figure A-1.

The design shown in figure A-1(a) proved impractical because the glass frit became plugged by the decomposing compounds. For this reason it was decided to use a tapered column.

The designs shown in figure A-1(b) and (c) permitted the oxide powder to run into the "T" and cause plugging. By extending the taper section and placing an undulation at the base of the column, plugging was eliminated. This design is shown in figure A-1(d). The sizes of the column shown in figure A-1(d) are optimized and identical to those used in most of the runs. Large orifices permitted oxide from the bed to run into the "T" and eventually cause plugging. (The 6-mm I. D. shown in figure A-1(d) prevented plugging.)

To minimize blowover, the column was expanded from 45-mm I. D. to 70-mm I. D. above the heated zone. Above the expanded section, an inverted Erlenmeyer flask was attached to the column as shown in figure 1.

From the analysis of tapered fluidized beds, the following generalizations were made:

(a) To obtain turbulent fluidization, a critical amount of powder (~ 7 g of MgO for a 2-inch-diameter column) must be used together with a critical flow rate of at least 2 liters of argon per minute. Somewhat higher flow rates (approximately 2.5 liters per minute) are needed for a 50/50 argon-hydrogen mixture.

(b) A vibrator was necessary to prevent sticking of the powders to the heated walls.

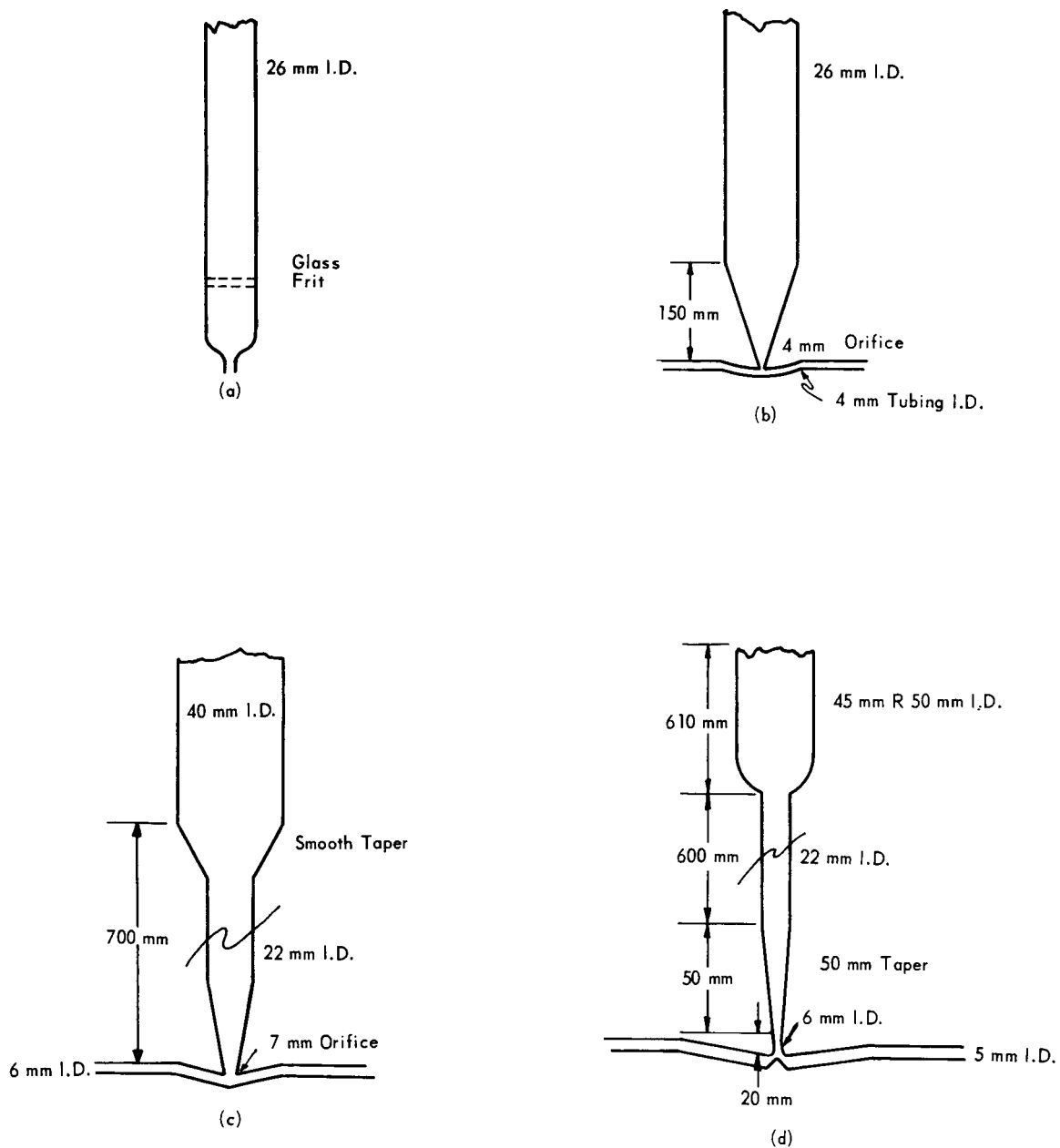


Figure A-1. Designs for Bottom Part of Column of Fluidized Bed

(c) Too large an orifice at the bottom permitted powder to enter the "T"; this resulted in plugging.

(d) Too narrow an orifice at the bottom caused the gas flow to channel through the bed instead of fluidizing the bed. The bed would ultimately collapse in this condition.

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