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DEVELOPMENT OF UNIDIRECTIONALLY SOLIDIFIED EUTECTIC CERAMIC MATRIX-METAL COMPOSITES

Final Report

Prepared under Contract N62269-75-C-0120

Naval Air Development Center
Warminster, Pennsylvania 18974

for

Naval Air Systems Command
Department of the Navy
Washington, D. C. 20361

by

C. O. Hulse

May 15, 1976

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The objective of this work was to identify and develop directionally solidified ceramic matrix-metal eutectic composites for use uncoated at temperatures ≥1315°C (2400°F) in aircraft gas turbines. The primary approach was to discover metal-oxygen systems in which the metal whisker phase would form a self-protective oxide coating when exposed to elevated temperatures. A number of metal-oxygen combinations were examined including Ta, Ta + Ta_2O_5, Ti or Nb with →

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Al_2O_3, Cr_2O_3 solid solutions, Cr and Cr + Ta with the $MgO \cdot Al_2O_3$ spinel, Cr with the mullite-zircon eutectic composition, Fe and Fe + Cr with the $FeO \cdot Cr_2O_3$ spinel with and without Al_2O_3 replacements for some of the Cr_2O_3. In addition the ternary monovariant eutectic trough wherein L → Al_2O_3, Cr_2O_3 + Cr, was determined for the Cr-Al_2O_3-Cr_2O_3 system which exhibited good resistance to oxidation at 1425°C (2600°F).

Although directional solidification experiments were run in tungsten crucibles prepared by chemical vapor deposition, it was not possible to obtain pore free, uniformly aligned eutectic microstructures in the Cr-Al_2O_3-Cr_2O_3 system. Similar experiments with ZrO_2 (Y_2O_3) additions with and without sapphire seeds indicated that the Al_2O_3, Cr_2O_3 solid solution matrix forces growth in a non-preferred direction for the reinforcement phases. This latter eutectic unseeded exhibited a weight gain of only ~3% after 52 hrs in static air at 1425°C (2600°F). The average strength in bending of this eutectic at 1540°C (2800°F) was 23,400 psi.

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Report R76-912084-4

Development of Unidirectionally Solidified Eutectic Ceramic Matrix-Metal Composites

FINAL REPORT
Contract N62269-75-C-0120

REPORTED BY Charles Hulse
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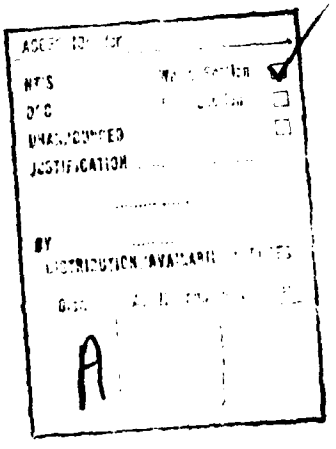
F. D. Lemkey, Principal Scientist
High Temperature Materials



Development of Unidirectionally Solidified Eutectic Ceramic Matrix-Metal Composites

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I. INTRODUCTION

This report describes the results for the second year of a research study directed toward the development of directionally solidified oxide matrix-metal eutectic materials for use in aircraft gas turbines. This work was supported by Naval Air Development Center contract N62269-75-C-0120, with Mr. I. Machlin of the Naval Air Systems Command as technical consultant and the Naval Air Development Center, Warminster, PA, as the contracting agency.

There are a number of circumstances which could produce improvements in the mechanical properties of ceramic-metal materials with a directional eutectic microstructure over that available from hot-pressed ceramics. An immediate possibility is that at high temperatures the minor metal phase will have the high strength characteristic of a material in whisker form and that this phase will directionally reinforce the somewhat ductile ceramic matrix in a typical composite manner. The reality of this possibility has been demonstrated in numerous metallic eutectic systems. Figure 1 shows that over a wide range of temperatures, similar Petch type equations relate strength to the spacing between phases in a eutectic (Ref. 1) just as they relate the strength to grain size in hot-pressed ceramics.

An important advantage of directionally solidified eutectics for high temperature strength applications is that their microstructure is extremely stable, practically to the melting point (Ref. 2). This stability results from the fact that their microstructures are produced directly from the molten state under conditions of thermodynamic equilibrium. If grain boundaries are present in these microstructures they are relatively few in number and generally parallel to the axis of primary reinforcement.

The strength of a eutectic composite at lower temperatures, where both phases may be more brittle, may be enhanced by a suitable selection of phases so that the matrix phase is placed in compression upon cooling due to differences in thermal expansion between the phases. A tensile stress applied to the bulk composite then will not result in a tensile stress in the continuous ceramic matrix phase until the compressive prestress is overcome. Tension is the primary failure mode of ceramic materials. The effectiveness of prestressing in increasing the strength of a ceramic matrix composite has been demonstrated (Ref. 3) and surface type prestressing is of considerable commercial importance in the glass industry. If, in addition to the prestress contribution, the matrix has a lower elastic modulus than the reinforcing phase, the amount of stress seen by the matrix phase during tensile loading of the composite will be even further reduced. A proportionately larger fraction of the applied stress is carried by the minor phase as the ratio of the modulus of reinforcing to matrix phase is increased.

The fracture of brittle materials is usually believed to involve the sudden growth of very fine flaws, called "Griffith Microcracks", which are always present in these materials. The importance of a flaw depends upon its size. Fracture normally involves the growth of the "critical" flaw which is the largest microcrack with the appropriate orientation to the applied load. Because the distribution of these flaws is random, the actual strengths of ceramics show a statistical distribution which is also a function of volume stressed. Some encouraging efforts have been made to limit the size of microcracks by the presence of a fine dispersion of second phase particles. If ceramic eutectics with a very fine, uniform microstructure can be produced, improved mechanical properties may be observed because the size of these microcracks are significantly limited.

The incorporation of a metal whisker phase into a ceramic should dramatically enhance the thermal conductivity and hence the resistance to thermal shock of the ceramic. A number of investigators (Refs. 4,5) have noted improvements in thermal shock resistance made by metal wire additions. Satisfactory resistance to thermal shock is a serious requirement for an aircraft gas turbine part.

The impact strengths of ceramics are generally so low that they are not usable in many important applications for which they might otherwise be well suited. The main mechanism by which energy is absorbed during fracture of brittle materials is through the production of new surfaces. The presence of a finely dispersed, high modulus fibrous or lamellar phase may function to deviate cracks and thus increase the amount of fracture surface produced during failure. These energies can be increased significantly if cracks are deflected so as to follow the dispersed phase-matrix interface. The Ni_3Al-Ni_3Cb intermetallic eutectic is an example where cracks are blunted and deflected along these interfaces rather than propagating in the usual brittle manner. An example of this delamination process is shown in Fig. 2. Although this material shows practically no tensile ductility at room temperature, it has a room temperature Charpy impact strength of about 1.75 ft-lbs for half-sized, notched samples.

In prior work sponsored by the Office of Naval Research at the United Technologies Research Center, it was demonstrated that directionally solidified oxide-oxide ceramic eutectics could exhibit exceptional strengths, particularly at elevated temperatures. Figure 3 shows flexural strength data for the Al_2O_3-ZrO_2 (Y_2O_3) eutectic at 1575°C (2865°F) compared with values obtained for a good commercial polycrystalline Al_2O_3 material at the same temperature (Ref. 6). The fracture toughness of this eutectic, as evaluated by the work-to-fracture technique, also showed significant increases over that required for single phase Al_2O_3.

Measurements of the work of fracture of directionally solidified MgO-CaO and ZrO_2-$CaO \cdot ZrO_2$ eutectics, both of which contain a relatively ductile oxide phase, showed dramatic increases in their resistance to fracture at elevated temperatures. Data for the ZrO_2-$CaO \cdot ZrO_2$ system are presented in Fig. 4. At elevated temperatures these two eutectics apparently behave in the classic composite manner wherein a ductile matrix phase is strengthened by a stronger reinforcement phase which is relatively much less ductile.

The incorporation of a metal phase which can also absorb energy by plastic flow can result in significant improvement in composite impact strength. In earlier studies at UTRC, the impact strengths of ceramic composites containing tungsten wires were examined. Charpy impact strengths of ~2 ft-lbs were measured for half-sized unnotched samples of mullite-tungsten wire composites with greater than 14 volume percent tungsten. In more recent work by Brennan (Ref. 7) at UTRC sponsored by the Naval Air Systems Command, the Charpy impact strength of hot-pressed Si_3N_4 was increased from 0.5 to 18.0 ft-lbs by the addition of 23 volume percent tantalum wires. Hart (Ref. 8) has reported that the Charpy impact strength of the Cr_2O_3-Mo eutectic is about 2.5 times that of fine grained Al_2O_3. He also reported a work-to-fracture energy for this eutectic of 40×10^4 ergs/cm^2 (.029 ft-lb/cm^2). This is approximately four times that of the Al_2O_3-ZrO_2 (Y_2O_3) eutectic shown in Fig. 3. Taken all together, the above fracture and impact data suggest that there is a considerable potential for improved toughness in ceramic matrix-metal eutectic systems.

Research during the first year of support for this program (Ref. 9) by the Naval Air Systems Command concentrated on a survey of various binary eutectic possibilities. The literature provided very limited information concerning metal-oxide phase equilibria. The primary intention was to identify metal-oxygen systems in which the metal whisker phase might form a self-protective oxide coating on exposure to air at elevated temperatures.

After some preliminary experiments with refractory metal strip heaters, the arrangement shown in Fig. 5 using a carbon susceptor for heating in an R.F. field was developed to make experimental melts for microstructural examination. During these melting experiments, an optical pyrometer was also used to sight directly on samples held in simple tungsten wire baskets to determine the temperatures of incipient and complete melting. The initial powders used for these samples were all of at least 99.9% purity and were hand mixed and isostatically cold pressed into rods at about 15,000 lbs/in.^{2} using evacuated rubber containers. Before melting, the samples were prefired in argon to about 1300°C (2370°F). Systems of interest were directionally solidified inside tungsten tubes prepared by chemical vapor deposition. The molten zone inside these tubes was produced by an external carbon ring susceptor in an R.F. field in an atmosphere of argon. Additives which provided additional oxygen to the melt such

as CeO_2, Cr_2O_3 and WO_3 were often necessary to increase the metal solubility sufficiently to form a continuous eutectic microstructure. A number of potentially interesting systems which responded well to directional solidification, Gd_2O_3-CeO_2-Ta, Y_2O_3-CeO_2-Ta and Y_2O_3-CeO_2-Y, could not survive a 24 hr exposure to air at 1093°C (2000°F). The Cr-Al_2O_3, Cr_2O_3 system, however, showed a surface attack of only a few mils after an exposure of 60 hrs in air at 1425°C (2600°F).

II. TECHNICAL PROGRESS

2.1 Melts Made With Tungsten Baskets Inside Carbon Susceptors

Melting experiments were made to determine the eutectic compositions along the pseudo-binary trough in the Cr-Al_2O_3-Cr_2O_3 ternary system. These compositions were of special interest because of the observation made during the first year's effort that eutectics in this system had good resistance to oxidation. Prefired sample slugs were melted inside a carbon susceptor in an argon atmosphere. The samples were held inside hand-made baskets made from 20 mil diameter W + 3% Re wire. As shown in Fig. 5, the samples could be continuously observed and their temperatures measured with an optical pyrometer through a quartz window as they were being heated. The temperatures of initial melting were indicated by a rounding-off of some sharp feature of the sample. Upon complete melting, the samples normally remained within the baskets because of surface tension forces.

Melting temperature data obtained for a fixed ratio between Al_2O_3 and Cr_2O_3 (0.7:0.3) for various additions of Cr metal is presented in Fig. 6. Similar data were obtained for other Al_2O_3:Cr_2O_3 ratios. Alumina and Cr_2O_3 show complete solubility in all proportions with each other (Ref. 10). The Cr-Cr_2O_3 system was also examined and the literature data (Refs. 11,12) confirmed. We determined the eutectic temperature and composition for the Cr-Cr_2O_3 system as being approximately 1685°C (3060°F) and 24 weight percent Cr compared with literature values of 1660°C (3020°F) and 21 weight percent Cr. Figure 7 shows the Cr-Cr_2O_3-Al_2O_3 ternary phase equilibrium diagram in argon using a carbon susceptor heater as determined by our measurements. The existence of a true eutectic between Cr and Al_2O_3 was not completely established. The melting temperature measured for the eutectic at 2030°C (3690°F) is close to that for pure Al_2O_3 and the Cr in the microstructure was widely dispersed, with the largest amounts being concentrated at grain boundaries.

In prior work (Ref. 6) it was shown that a eutectic with outstanding high temperature strength existed between Al_2O_3 and ZrO_2 stabilized with Y_2O_3. Melting experiments determined that the eutectic melting temperature for this oxide was ~1870°C (3400°F). Additions of Cr to this eutectic together with a partial replacement of some of the Al_2O_3 with Cr_2O_3 resulted in the discovery of a ternary eutectic at approximately the following weight percentages: 24 Cr, 23.5 Al_2O_3, 23.5 Cr_2O_3, 25 ZrO_2 and 4 Y_2O_3. This eutectic melted at approximately 1662°C (3020°F) and consisted of whiskers of Cr and ZrO_2 stabilized in the cubic structure by the Y_2O_3, uniformly dispersed in a matrix of Al_2O_3 and Cr_2O_3 in solid solution.

A general summary of all of the melting experiments is presented in Table I. A variety of other melt additions were made to the basic Cr-Al_2O_3-Cr_2O_3 system. These included additions of Ta and Fe, and replacement of the Cr by Ta or by mixtures of Ta with Ta_2O_5. Replacement of Cr by Ta resulted in porosity and evidence of an additional oxide phase. In other melts there was evidence of reaction with the tungsten wire basket and good metal solubility. Evidence for good solubility of metal in the melt was assumed when the metal phase was uniformly dispersed on a fine scale in a polished section. At the other extreme, in a melt with poor solubility the metal was concentrated in a few large spheres or as irregular globs. Additions of Ta and Cr resulted in uneven metal solubility and a rod-like eutectic was seen. In other melts, porosity was an evident problem as well as the presence of an additional oxide phase. Additions of the $TaCr_2$-Cr eutectic to Al_2O_3-Cr_2O_3 solid solutions also resulted in isolated areas of good eutectic microstructures, a metal-metal eutectic and a sharply defined additional grain boundary phase. Melts with additions of Fe to the Cr resulted in extremely fine metal dispersions which could indicate either a eutectic or a solid state precipitation process.

Melts of Ti and Nb with Al_2O_3 and Cr_2O_3 showed porosity, poor metal solubility and contained additional oxide phases. These are probably suboxide phases which would not be strong or stable in an air environment at elevated temperatures.

Melts of the MgO-$MgO\cdot Al_2O_3$ and $MgO\cdot Al_2O_3$-Al_2O_3 eutectic compositions did not show evidence of reaction with W wires whereas reactions were evident with Cr additions. In both cases there was evidence of metal solubility but regular eutectic microstructures were not seen.

A mullite-zircon eutectic melt had a structure filled with long dendrites which appeared to have decomposed to form extremely regular eutectic-like structures at right angles to the direction of solidification. An example of this microstructure is shown in Fig. 8. The areas between the dendrites appeared to be glass. Chromium metal was not soluble in this melt.

Additions of Ta and Cr to the $MgO\cdot Al_2O_3$ spinel resulted in poor metal solubility except for the ternary eutectic present in the grain boundaries.

Attempts to melt Fe with the $FeO\cdot Cr_2O_3$-Cr_2O_3 eutectic composition resulted in extensive reaction with the tungsten basket. The problem was less with melts made in Al_2O_3 or $Al_2O_3\cdot MgO$ crucibles. Additions of both Fe and Cr to $FeO\cdot Cr_2O_3$-Cr_2O_3 also resulted in reaction with the W baskets. Reaction was less of a problem when MgO or Al_2O_3 crucibles were used. A metal-oxide eutectic was observed in the grain boundaries of these melts together with good metal solubilities. Figure 9 shows the typical microstructure observed in a melt of

Cr_2O_3 and Fe_2O_3 combined with a mixture of Fe and Cr showing good metal solubility. In other melts inside short tubes of CVD tungsten, porosity and extensive metal solubility throughout was observed as well as reaction with the W.

Melts of Cr with the $FeO \cdot Cr_2O_3$-Cr_2O_3 eutectic composition in short CVD tungsten tubes revealed two phase metal globs and areas of metal-oxide eutectic. The oxide phase showed a tendency either to fine scale porosity or pitting during the polishing operation.

2.2 Directional Solidification in Refractory Metal Tubes

Directional solidification experiments were conducted using as containers commercial CVD tungsten tubes*, 4 in. long x 3/8 in. I.D. with a 0.005 in. thick wall. The tubes fitted on the end of a refractory metal post and were traversed by this support down through a carbon ring susceptor which created a zone of molten eutectic within the tube. A schematic of the apparatus is shown in Fig. 10. The bottom of the containment tube was self-sealed by the first melt which solidified on or near the refractory metal support post. The upper end of the tube was loosely sealed with a refractory metal cap. The use of a container was considered desirable because it corresponds more closely, than does a floating molten zone approach, to current foundry practice. It had the addition advantage that it tended to inhibit the loss of constituents by evaporation.

It became apparent that longer containment tubes would be desirable to make ingots of good size. Attempts to use less costly tantalum tubes were abandoned because of their reactivity. Longer ingots were made successfully using two 4 in. long tungsten tubes on top of one another held in position by several turns of Mo foil fastened with tungsten wire. An internal capability to prepare 3/8 in. I.D. x 7 in. long tungsten tubes by chemical vapor deposition was then developed under Corporate sponsorship. These tubes were used for the remainder of the program after some preliminary attempts to use tantalum tubes coated internally with CVD tungsten were unsuccessful.

A major problem in making sound ingots was the elimination of the gases evolved during melting. These gases when entrapped would result in the formation of a number of short ingot sections with air pockets in between and/or bursting of the tungsten tubes. The use of a vibrator of variable force and frequency at the bottom of the refractory metal support rod was of some help but the best solution was to apply a weight of about 90 gms at the top of the ceramic-metal charge rod and to use smaller feed rods (i.e. 1/16 in. diameter). Small

*Ultramet, Paccima, CA

bleed holes in the upper sections of the tungsten tubes were also found to be helpful for the relief of gas pressures.

A general summary of all of the directional solidification experiments is presented in Table II. Many directionally solidified ingots of the Cr-Al_2O_3-Cr_2O_3 eutectic system were prepared. Most ingots had many small sections with relatively good structure as shown, for example, in Fig. 11. Obtaining a uniformly good microstructure throughout was difficult. At speeds greater than about 1 cm/hr the structure became cellular and it became especially difficult to obtain a uniform microstructure. The cells tended to be divided into three sections parallel to the direction of solidification in a similar manner as was observed in the Al_2O_3-ZrO_2 (Y_2O_3) eutectic system (Ref. 6). All the whiskers in a section tended to grow at the same large angle to the direction of solidification. The second major difficulty with this system was the presence of fine porosity especially in cell boundaries. This problem was probably due to the volatility of the Cr constituent. The ingots appeared to be pore-free on the macroscale and were easily separated from the tungsten containment tubes. Although we were not able to prepare material with uniform, pore-free microstructures, three unnotched Charpy impact samples were prepared and tested at room temperature. The average impact strength obtained was 0.01 ft-lb.

Directionally solidified ingots of the three phase eutectic, 24 w/o Cr, 23.5 Al_2O_3, 23.5 Cr_2O_3, 35 ZrO_2 and 4 Y_2O_3 mentioned earlier, were also prepared. This eutectic had a microstructure similar to those observed in the original oxide-oxide eutectic (Ref. 6). Whiskers of ZrO_2 stabilized with Y_2O_3 grow in a matrix of Al_2O_3, Cr_2O_3 solid solution with the chromium metal associated primarily with the zirconia phase.

Plane front solidification was only observed at solidification speeds below ~0.5 cm/hr. Figure 12 shows a longitudinal view of the microstructure obtained at a solidification speed of 0.5 cm/hr. The ZrO_2 phase is very irregular while the metal phase shows no directionality. The Cr phase is associated primarily with the ZrO_2 (Y_2O_3) phase. The C-axis of the Al_2O_3, Cr_2O_3 solid solution and the [111] axis of the stabilized ZrO_2 are parallel to the direction of solidification. The orientation of the Cr phase has not been determined. Electron microprobe experiments confirmed that the metal phase was pure Cr with only trace amounts of tungsten present. Powder X-ray experiments confirmed that all the zirconia present had the cubic crystal structure.

In earlier studies of oxide eutectics it was observed that the ZrO_2 whiskers preferred to grow at a large angle to the liquid solid interface. Very perfect microstructures were obtained when the eutectic was grown from a properly oriented seed. Figure 13 shows in transverse section the perfect whisker microstructure that can be obtained with the metal whisker reinforced eutectic when the Al_2O_3,

Cr_2O_3 phase is forced to grow parallel to the $[02\bar{2}4]$ by use of a sapphire seed crystal. In this picture the small whiskers are ZrO_2 (Y_2O_3), the larger irregular cross-section whiskers are Cr metal and the matrix is Al_2O_3, Cr_2O_3 in solid solution.

The results of experiments conducted to measure the oxidation resistance of the unseeded "ternary" eutectic are shown in Fig. 14. After an initial oxidation of the exposed metal whiskers, there was practically no change in the weight of this material in static air at 1425°C (2600°F).

The strengths of four samples of the unseeded "ternary" eutectic grown at 1.2 cm/hr were measured in bending at 1540°C (2800°F) in argon. The average strengths were 23,400 lbs/in.^{2}. The samples were brittle and there were jogs in the loading record suggesting cracking well below the ultimate strength. After testing the samples were all observed to contain significant longitudinal cracks. Many cracks were also observed in the sample used to obtain the oxidation data presented in Fig. 14. Apparently this material contains appreciable internal stresses which can be aggravated by heating.

A small series of solidification experiments were also run in which Fe was present together with Fe_2O_3 as additions to the Cr-Al_2O_3-Cr_2O_3 ternary. The results, i.e. reaction, suggest that tungsten crucibles cannot be used to contain melts with Fe additions.

III. SUMMARY AND CONCLUSIONS

The phase equilibria diagram for the Cr-Cr_2O_3-Al_2O_3 system has been determined for melts made in argon inside a carbon susceptor.

Many metal-oxide melts were made primarily in tungsten wire baskets in search of controllable, regular eutectic microstructures. Many systems showed evidence of extensive metal solubility in the melts and in some case regular eutectic structures were observed. This appeared to be particularly true when the matrix was a spinel capable of a variable valence.

It was not possible to directionally solidify the Cr-Al_2O_3-Cr_2O_3 ternary eutectic with uniform microstructures free of fine scale porosity.

A multicomponent eutectic of stabilized zirconia and chromium whiskers in an Al_2O_3, Cr_2O_3 solid solution matrix could be seeded with sapphire to grow with very perfect microstructures. However, this eutectic tended to develop cracks when heated in air to elevated temperatures.

The multicomponent eutectic showed bend strengths of 23,400 lbs/in.^{2} at 1540°C (2800°F) in argon and a weight gain of about 3% after 52 hrs in air at 1425°C (2600°F).

IV. FUTURE WORK

The possibility of forming regular oxide-metal eutectics containing NiAl as the metal phase will be examined. This metal compound is relatively refractory (M.P. 1735°C) and it should have excellent resistance to oxidation.

The search for oxide matrix candidates will concentrate on the various spinel compounds. These have cubic crystal structures which should also favor regular eutectic microstructures. They also can have a variety of valence states which should be favorable to metal solubility in the melt.

Because of recent difficulties with container-melt reaction, directional solidification runs will be made without containers using the floating molten zone technique.

The effects of small partial pressures of oxygen and neutral atmospheres on metal solubility in the melt will be examined. A Mo ring susceptor with Ta heat shields has been used successfully to reach 1800°C (3270°F) in argon but it has not been used as yet for melting eutectics.

V. ACKNOWLEDGEMENTS

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Table I

Melting Runs in Tungsten Baskets

(Argon Atmosphere, Carbon Susceptor)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-75-34	53 Cr_2O_3 25 Al_2O_3 22 Cr	Two samples run, both melting at 1754° corrected, molten 1808° corrected.
W-Mp-75-35	53.5 Cr_2O_3 22.35 Al_2O_3 24.15 Cr	Completely molten at 1795° corrected.
W-Mp-75-36	55.2 Cr_2O_3 22.99 Al_2O_3 21.85 Cr	Melting 1753° corrected.
W-Mp-75-37	38.5 Cr_2O_3 38.5 Al_2O_3 23 Cr	Melting 1732° corrected, molten 1795° corrected.
W-Mp-75-38	37.35 Cr_2O_3 37.35 Al_2O_3 25.3 Cr	Melting 1743° corrected, molten 1795° corrected.
W-Mp-75-39	39.65 Cr_2O_3 39.65 Al_2O_3 20.7 Cr	Completely molten 1795°C corrected.
W-Mp-75-40	53.5 Cr_2O_3 22.35 Al_2O_3 24.5 Cr	Molten 1764°C corrected.
W-Mp-75-41	39.65 Cr_2O_3 39.65 Al_2O_3 20.7 Cr	Basket came loose, run lost, repeated: melting 1737° corrected, molten 1769° corrected.
W-Mp-75-42	53.5 Cr_2O_3 22.35 Al_2O_3 24.15 Cr	Melting 1740° corrected, molten 1758° corrected.
W-Mp-75-43	42 Cr_2O_3 42 Al_2O_3 16 Cr	Melting 1753° corrected, molten 1764° corrected.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (^{o}C)
W-Mp-75-44	41 Cr_2O_3 41 Al_2O_3 18 Cr	Melted 1722° corrected, no further melting at 1748° corrected.
W-Mp-75-45	37 Cr_2O_3 37 Al_2O_3 26 Cr	Melting at 1753° corrected.
W-Mp-75-46	52.99 Cr_2O_3 22.01 Al_2O_3 25 Cr	Melting 1753° corrected
W-Mp-75-47	52.28 Cr_2O_3 21.72 Al_2O_3 26 Cr	Melting 1764° corrected, molten 1774° corrected.
W-Mp-75-48	50.87 Cr_2O_3 21.13 Al_2O_3 28.0 Cr	Melting 1753° corrected, molten 1790° corrected.
W-Mp-75-49	37 Cr_2O_3 37 Al_2O_3 26 Cr	Melting 1690° obs, 1753° corrected, molten 1710-20 obs, 1774°-85° corrected.
W-Mp-75-50	52.99 Cr_2O_3 22.01 Al_2O_3 25.0 Cr	Melting 1753° corrected, molten 1785° corrected.
W-Mp-75-51	55.2 Cr_2O_3 22.99 Al_2O_3 21.85 Cr	Melting 1753° corrected, molten 1790° corrected, repeated, same results.
W-Mp-75-52	42 Cr_2O_3 42 Al_2O_3 16 Cr	Melting 1753° corrected, molten 1774°.
W-Mp-75-53	41 Cr_2O_3 41 Al_2O_3 18 Cr	Melting 1743° corrected, molten 1764° corrected.
W-Mp-75-54	37 Cr_2O_3 37 Al_2O_3 26 Cr	Melting 1743° corrected, molten 1774° corrected.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (^{o}C)
W-Mp-75-55	53 Cr_2O_3 22 Al_2O_3 25 Cr	Melting 1753° corrected, molten ~ 1669° - 1774° (not completely molten when removed).
W-Mp-75-56	76 Cr_2O_3 13.5 Al_2O_3 10.0 Cr	Melting 1922° corrected, completely molten 1935 - 1940° corrected.
W-Mp-75-57	73 Cr_2O_3 12.9 Al_2O_3 14.0 Cr	Melting 1884° corrected, molten 1902° corrected.
W-Mp-75-58	71.4 Cr_2O_3 12.6 Al_2O_3 16.0 Cr	Melting 1930° corrected, molten 1944° corrected.
W-Mp-75-59	93 Al_2O_3 7 Cr	No melting up to 1955° corrected. Furnace problems, run stopped, repeated, melting 2050° corrected, molten 2075° corrected.
W-Mp-75-60	90 Al_2O_3 10 Cr	Melting 2040° corrected, molten 2044° corrected.
W-Mp-75-61	87 Al_2O_3 13 Cr	Melting 2075° corrected, molten 2085° corrected.
W-Mp-75-62	45 Al_2O_3 5 Cr_2O_3 42.5 ZrO_2 7.5 Y_2O_3	Melted 1856° corrected, repeated. Melting 1870° corrected, molten 1890° corrected.
W-Mp-75-63	5.456 Cr_2O_3 49.0 Al_2O_3 38.66 ZrO_2 6.84 Y_2O_3	Melting 1860° corrected, molten 1870° corrected.
W-Mp-75-64	4.0 Cr_2O_3 36.0 Al_2O_3 51.0 ZrO_2 9.0 Y_2O_3	Melting 1880° corrected, molten 1930° corrected.
W-Mp-75-65	36.83 Al_2O_3 4.09 Cr_2O_3 28.96 ZrO_2 5.13 Y_2O_3 25.0 Cr	Melting 1808° corrected, molten 1830° corrected.

Table I (Cont'd)

Run#	Material (Wt %)	Remarks (°C)
W-Mp-75-66	39.28 Al_2O_3 4.36 Cr_2O_3 30.93 ZrO_2 5.47 Y_2O_3 20 Cr	Melting 1812° C corrected, molten 1875° corrected
W-Mp-75-67	41.74 Al_2O_3 4.64 Cr_2O_3 32.86 ZrO_2 5.18 Y_2O_3 1.5 Cr	Melting 1812° corrected, molten 1848° corrected. Sample had pink color after melting
W-Mp-75-68	10 Ta 76 Al_2O_3 13.5 Cr_2O_3	Melting 1935° corrected, molten 1950° corrected
W-Mp-75-69	14 Ta 73.1 Al_2O_3 12.9 Cr_2O_3	Melting 1925° corrected, molten 1940° corrected
W-Mp-75-70	16 Ta 73.1 Al_2O_3 12.9 Cr_2O_3	Melting 1925° corrected, molten 1940° corrected
W-Mp-75-70	16 Ta 71.4 Al_2O_3 12.6 Cr_2O_3	Melting 1892° corrected, molten 1930° corrected
W-Mp-75-71	9.5 Ta 2.5 Cr 76.5 Al_2O_3 13.5 Cr_2O_3	Melting 1881° corrected, molten 1930° corrected
W-Mp-75-72	13.3 Ta 3.5 Cr 73.1 Al_2O_3 12.9 Cr_2O_3	Melting 1890° corrected, molten 1920° corrected

Table I (Cont'd)

Run#	Material (Wt %)	Remarks (°C)
W-Mp-75-73	15.2 Ta 4.0 Cr 71.4 Al_2O_3 12.6 Cr_2O_3	Melting 1920-1930° corrected, molten 1940° corrected; rapid melting once started
W-Mp-75-74	5 Cr 95 Al_2O_3	Melting 2050°, molten 2060° corrected
W-Mp-75-75	8 Cr 85 Al_2O_3	Melting 2040°, molten 2050° corrected
W-Mp-75-76	11 Cr 89 Al_2O_3	Melting 2040°, molten 2050° corrected
W-Mp-75-77	15 Cr 85 Al_2O_3	Melting 2035°, molten 2060° corrected
W-Mp-75-78	12 Cr 23.76 Al_2O_3 23.76 Cr_2O_3 34.4 ZrO_2 6.1 Y_2O_3	Melting 1645°, molten 1670° corrected. Most of melt lost when basket fused to insulation
W-Mp-75-79 (5/28/75)	16 Cr 22.68 Al_2O_3 22.68 Cr_2O_3 32.84 ZrO_2 5.80 Y_2O_3	Melting 1650°, molten 1670° corrected
W-Mp-75-80	23 Cr 20.78 Al_2O_3 20.78 Cr_2O_3 30.13 ZrO_2 5.32 Y_2O_3	Melting 1670-1680°, molten 1730 corrected.
W-Mp-75-81	28 Cr 19.44 Al_2O_3 19.44 Cr_2O_3 28.15 ZrO_2 4.97 Y_2O_3	Melting 1662°, molten 1690° corrected

Table 1 (Cont'd)

Run#	Material (Wt %)	Remarks (°C)
W-Mp-75-82	14 Ta 43 Al_2O_3 43 Cr_2O_3	Melting 1680°, molten 1750° corrected. Repeated 1690° melting, 1742° molten, not clear that melting really occurred (sintering)
W-Mp-75-83	14 Ta 20 Ta_2O_5 23 Al_2O_3 23 Cr_2O_3	Melting 1585°, molten 1625° corrected
W-Mp-75-84	14 Ta 40 Ta_2O_5 23 Al_2O_3 23 Cr_2O_3	Melting 1570°, molten 1580° corrected
W-Mp-75-85	14 Ta 40 Ta_2O_5 46 Cr_2O_3	Melting 1540°, molten 1553° corrected
W-Mp-75-86	10 Cr 90 Cr_2O_3	Melting 1657°, molten 1772°, corrected. Melted, but edges still sharp, do over.
W-Mp-75-87	15 Cr 85 Cr_2O_3	Melting 1690°, molten 1731° corrected.
W-Mp-75-88	20 Cr 80 Cr_2O_3	Melting 1657°, molten 1708° corrected. Close to eutectic but oxide rich.
W-Mp-75-89	25 Cr 75 Cr_2O_3	Melting 1670°, molten 1700°, corrected
W-Mp-75-90	30 Cr 70 Cr_2O_3	Melting 1690-1700°, molten 1710°-1720 Much eutectic, metal dendrites and a few oxide dentrites. Guess eutectic ~25 w/o Cr.
W-Mp-75-91	Al_2O_3 sintered powder	Melting 1785°, 1989° - do over - don't use this data.
W-Mp-75-92	5 Cr 95 Cr_2O_3	Completely molten 2050° corrected. Some metal, extremely dispersed.

Table I (Cont'd)

Run#	Material (Wt %)	Remarks (°C)
W-Mp-75-93	8 Cr 92 Cr_2O_3	Melting 2020°C, molten 2050° corrected. Some eutectic like structure, metal better dispersed than 5 w/o Cr.
W-Mp-75-94	10 Cr 92 Cr_2O_3	Melting 1774°, molten 1845°. Very little eutectic, oxide grains with metal in boundaries.
W-Mp-75-95	100 % Al_2O_3 Sintered powder	1790° sintering, molten 2062°, corrected
W-Mp-75-96	5 Nb 47.5 Al_2O_3 47.5 Cr_2O_3	Melting 1987°, molten 1998°C, corrected. Sample fused to heater, repeated melting 1970°, molten 1996°, corrected.
W-Mp-75-97	10 Nb 45 Al_2O_3 45 Cr_2O_3	Melting 1755°, molten 1808°, corrected. Repeated 1737° melting, 1785° molten Final sample very porous.
W-Mp-75-98	15 Nb 42.5 Al_2O_3 42.5 Cr_2O_3	Melting 1732°, molten 1758°C corrected. Final sample is more molten than -97, still some porosity (roughness in lower sections).
W-Mp-75-99	5 Ta 47.5 Al_2O_3 47.5 Cr_2O_3	Melting 1753°, molten 1811° corrected. Melted well but porous.
W-Mp-75-100	10 Ta 45 Al_2O_3 45 Cr_2O_3	Melting 2063°, molten 2073° corrected. Melted fragments below basket appear as solid globs of melt with no porosity. Repeat with smaller charge, tighter basket.
W-Mp-75-101	15 Ta 42.5 Cr_2O_3 42.5 Al_2O_3	Melting 1785°, molten 1808°, porous, do over with smaller sample.
W-Mp-75-102	5 Ti 47.5 Al_2O_3 47.5 Cr_2O_3	Melting 1720°, molten 1742°. Nice melting compared with -101 but porous.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-75-103	45 Al_2O_3 45 Cr_2O_3 10 Ti	Molten 1690°C, fused to heater, shut down, repeated, melting 1660°, molten 1678°, quite porous, 2 oxide phases, metal not diffused, poor polishing.
W-Mp-75-104	42.5 Al_2O_3 42.5 Cr_2O_3 15 Ti	Melting 1665°, molten 1700°, porous, poor to polish, similar to -103.
W-Mp-75-105	45 Al_2O_3 45 Cr_2O_3 10 Ta	Sintering 2010, molten 2020°.
W-Mp-75-106	42.5 Al_2O_3 42.5 Cr_2O_3 15 Ta	Melting 1755°, molten 1820°. Some area of interaction with W wires, second oxide at grain boundaries (lighter) with eutectic in it. Good metal solution - need more metal.
W-Mp-75-107	45 Al_2O_3 45 Cr_2O_3 10 Ta	Melting 2010°, molten 2062°.
W-Mp-75-108	45.2 Al_2O_3 45.2 Cr_2O_3 15 Ta	Melting 1715°, molten 1762°, sample very porous, poor polish, two oxides with primary or undissolved primary.
W-Mp-75-109	90 Cr_2O_3 10 Cr	Melting 2010°, molten 2020°, metal dispersed, primarily in G.B.
W-Mp-75-110	88 Al_2O_3 12 Cr	Melting 2015°, molten 2025°.
W-Mp-75-111	85 Al_2O_3 15 Cr	Melting 2030°, molten 2041°, very dense (no pores), definite indications of eutectic, long whiskers especially at boundaries-need more metal.
W-Mp-75-112	79 Cr_2O_3 21 Cr	Melting 1690°, molten 1700°.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-75-113	76 Cr_2O_3 24 Cr	Melting 1672°, molten 1700°.
W-Mp-75-114	65 Cr_2O_3 35 Cr	Melting 1670°, molten 1691°, good eutectic structures, best ~22 w/o. Porosity but not excessive.
W-Mp-75-115	44 Al_2O_3 44 Cr_2O_3 12 Nb	Melting 1720°, molten 1738°, dark (transparent) primary grains, boundaries oxide-oxide (sub) eutectic and metal-oxide (sub) eutectic.
W-Mp-75-116	43.25 Al_2O_3 43.25 Cr_2O_3 13.5 Nb	Molten 1710°, some melt dropped from basket, structures like -115.
W-Mp-75-117	41 Al_2O_3 41 Cr_2O_3 18 Nb	Melting 1720°, molten 1741°, 3 phases, no sign of eutectics.
W-Mp-75-118	44.5 Al_2O_3 44.5 Cr_2O_3 11 Cr	Melting 1732°, molten 1753°.
W-Mp-75-119	44 Al_2O_3 44 Cr_2O_3 12 Cr	Melting 1690°, molten 1725°.
W-Mp-75-120	43.5 Al_2O_3 43.5 Cr_2O_3 13 Cr	Melting 1700°, molten 1732°, metal poor, good eutectic structure but with porosity. Structures better than -118 & -119. Evidence of reaction with W.
W-Mp-75-121	74.8 Al_2O_3 13.2 Cr_2O_3 12 Cr	Melting 1832°, molten 1895°.
W-Mp-75-122	73.95 Al_2O_3 13.05 Cr_2O_3 13 Cr	Melting 1825°, molten 1880°.

Table I (Cont'd)

Exp. #	Material (Wt %)	Remarks (°C)
W-Mp-75-123	44.5 Al_2O_3 44.5 Cr_2O_3 11 Cr	Melting 1737°, molten 1764°.
W-Mp-75-124	44 Al_2O_3 44 Cr_2O_3 12 Cr	Melting 1732°, molten 1764°.
W-Mp-75-125	44 Al_2O_3 44 Cr_2O_3 12 Cr	Melting 1725°, molten 1753°.
W-Mp-75-126	43.5 Al_2O_3 43.5 Cr_2O_3 13 Cr	Melting 1705°, molten 1732°.
W-Mp-75-127	80 Cr_2O_3 20 Cr	Partly melted 2030°.
W-Mp-75-128	75 Al_2O_3 25 Cr	Melting 2010°, molten 2030°, fine dispersion of metal, no regular structure. Metal associated mostly with G.B., oxide difficult to polish without pits depending upon orientation.
W-Mp-75-129	55 Al_2O_3 45 MgO	Molten 2010°, very close to eutectic, no evidence of reaction with W (MgO-spinel).
W-Mp-75-130	46.75 Al_2O_3 38.25 MgO 15 Cr	Melting 2000°, molten 2020°, (MgO-spinel eutectic). Cr metal dispersed but not regular, long Cr whiskers, significant attack on W by melt.
W-Mp-75-131	97.3 Al_2O_3 2.7 MgO	Molten 2010°, difficult to see, apparently off eutectic, lots of dendrites.
W-Mp-75-132	82.7 Al_2O_3 2.3 MgO 15 Cr	Melting 1965°, molten 1975°, metal dispersed but no regular structure. Can't see Al_2O_3-spinel eutectic, many pits during polishing.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-75-133	38.3 Al_2O_3 31.7 ZrO_2 30 SiO_2	Melting 1710°, molten 1773°, mullite-zircon composition. Structure filled with dendrites which have "decomposed" to eutectic-like structures, apparent glass between dendrites.
W-Mp-75-134	32.55 Al_2O_3 26.95 ZrO_2 25.5 SiO_2 15 Cr	Mullite-zircon-Cr, metal not very soluble, not dispersed, where it appears there are dendrites of an additional darker phase ($SiO_2 \cdot Cr_2O_3$?)
W-Mp-75-135	38 Al_2O_3 38 Cr_2O_3 8 Ta 6 Cr	Melting 1680°, molten 1715°, suggestion of 2nd oxide phase. Rod-like eutectic, metal incompletely dissolved. Second oxide may be same phase of different orientation which polishes differently.
W-Mp-75-136	76 Al_2O_3 8 Ta 16 Cr	Melting 1930°, molten 1955°, degenerate eutectic (?). Additional phase or "chip-outs" during polishing.
W-Mp-75-137	76 Al_2O_3 16 Cr 8 Ta	Melting 1930°, molten 1955°, excess metal, some rod-like eutectic, poor polish.
W-Mp-75-138	27.5 Al_2O_3 27.5 Cr_2O_3 15 Ta 30 Cr	Melting 1690°, molten 1710°, excess metal, not well dispersed. Some rod-like eutectic, 2 oxide phases, metal globs 2 phase.
W-Mp-75-139	38.5 Al_2O_3 38.5 MgO 20.1 Cr 9.9 Ta	Melting 1950°, molten 1975°, rod-like eutectic (metal whiskers) in between acicular crystals, metal not well dispersed. Metal globs 2-phase. System looks worthy of further effort.
W-Mp-75-140	38 Al_2O_3 38 Cr_2O_3 18 Ta 6 Cr	Melting 1690°, molten 1715°.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-75-141	27.5 Al_2O_3 27.5 Cr_2O_3 33.75 Ta 11.25 Cr	Melting 1580°, molten 1615°, porous, poor metal solubility.
W-Mp-75-142	38.5 Al_2O_3 31.5 MgO 9.9 Ta 20.1 Cr	Very dense, transparent, primary phase (dark) in excess, metal solubility poor except for good ternary eutectic in grain boundaries. System worthy of further investigation.
W-Mp-76-7	81 Cr_2O_3 14.1 Fe_2O_3 4.9 Fe	Attempt to make $FeO \cdot Cr_2O_3$-Cr_2O_3 eutectic, at 1800°C reaction with W basket.
W-Mp-76-8	24 Al_2O_3 54 Cr_2O_3 15 Cr 7 Ta	Attempt Cr_2O_3-Al_2O_3 S.S. with $TaCr_2$-Cr eutectic. Melted 1638°, globs of metal, semi-dendritic; grains contain some good eutectic, others clear, a sharply defined grain boundary phase appears to be unscratched. In some area this phase is dispersed, may cause poor polish. Is Ta_2O_5 present?
W-Mp-76-9	21.5 Al_2O_3 48.5 Cr_2O_3 20 Cr 10 Ta	Melted 1650°, globs of semi-dendritic metal, cannot see metal-metal eutectic. Grain boundary phase as in -8.
W-Mp-76-10	81 Cr_2O_3 14.1 Fe_2O_3 4.9 Fe	Repeat of -7, material fell through W basket above 1800°C, did not melt.
W-Mp-76-11	62.4 Cr_2O_3 10.8 Fe_2O_3 3.8 Fe 23 Cr	Material reacted with W basket at 1750°C.
W-Mp-76-12	62.4 Cr_2O_3 10.8 Fe_2O_3 3.8 Fe 23 Cr	Melted in MgO crucible at 1960°, "Eutectic" metal-ceramic in grain boundaries.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-76-13	62.4 Cr_2O_3 10.8 Fe_2O_3 11.8 Fe 15.0 Cr	Melted 1800°C in alumina crucible, not much reaction although crucible leaked, microstructure similar to -12.
W-Mp-76-14	62.4 Cr_2O_3 10.8 Fe_2O_3 3.8 Fe 15.5 Cr 7.5 Ta	Reacted with alumina crucible (boiling?) at ~1760°C.
W-Mp-76-15	20.6 Al_2O_3 42.4 Cr_2O_3 3.8 Fe 10.8 Fe_2O_3 23 Cr	Good melt, some reaction with ZrO_2 crucible, many interesting structures. Melted 1730°, oxide-oxide eutectic looks like Y_2O_3-ZrO_2 solid state reaction, considerable metal solubility.
W-Mp-76-16	20.6 Al_2O_3 42.4 Cr_2O_3 10.8 Fe_2O_3 15.0 Cr 11.8 Fe	Melted 1800°, reaction and some melt through of crucible.
W-Mp-76-17	30.8 Cr_2O_3 46.2 Fe_2O_3 23.0 Cr	Did not melt completely at 1840°, slight reaction with spinel crucible.
W-Mp-76-18	15.4 Cr_2O_3 61.6 Fe_2O_3 23 Cr	Melted through bottom of alumina crucible at 1800°.
W-Mp-76-19	21.5 Al_2O_3 48.5 Cr_2O_3 20 Cr	Melted through bottom of alumina crucible at 1680°.
W-Mp-76-20	21.5 Al_2O_3 48.5 Cr_2O_3 10 Cr 20 Fe	Melted through bottom of ZrO_2 crucible at 1750°.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-76-21	21.5 Al_2O_3 49 Fe_2O_3 6 Cr 24 Fe	Melted through bottom of ZrO_2 crucible at 1720°.
W-Mp-76-22	62.4 Cr_2O_3 10.8 Fe_2O_3 3.8 Fe 15.5 Cr 7.5 Ta	$FeO \cdot Cr_2O_3$-Cr_2O_3 eutectic + Cr-Cr_2Ta eutectic, partial melt in 1 3/8" bond CVD tungsten tube; metal globs, fine dispersion of metal in eutectic only near end. Much evidence of sharp-sided grain boundary phase, may disperse to form "microporosity" on polishing.
W-Mp-76-23	62.4 Cr_2O_3 10.8 Fe_2O_3 11.8 Fe 15.0 Cr	Repeat of -13 with tungsten; melted at 1880°, good metal solubility but metal rich. Some reaction with W. Matrix rough, perhaps due to spinel formation. Metal globs 2-phase.
W-Mp-76-24	81 Cr_2O_3 41.1 Fe_2O_3 4.9 Fe	Repeat of -10 in W, melted well at ~1840°, some reaction with W. Two phase metal globs + pores. Grey, sharp edged phase again at boundaries, primary grains filled with chips or pits. Conclude Fe not all converted to $FeO \cdot Fe_2O_3$.
W-Mp-76-25	42.4 Cr_2O_3 20.0 Al_2O_3 10.8 Fe_2O_3 11.8 Fe 15.0 Cr	Repeat of -16 in W, good melt at 1890°, some liquid lost through crucible. Considerable porosity, metal globs, considerable metal solubility on a fine scale, some large grains of completely dense oxide.
W-Mp-76-26	30.8 Cr_2O_3 46.2 Fe_2O_3 23.0 Cr	Repeat of -17 in W, Fe_2O_3, Cr_2O_3 + Cr, melting at ~2000°, 2-phase metal S.S. globs, some metal dispersed well, good eutectic in one well melted area. Oxide tends to fine scale pits or porosity.
W-Mp-76-27	15.4 Cr_2O_3 61.6 Fe_2O_3 23 Cr	Repeat of -18 in tungsten, melted at ~1890°, some melt lost through W. Microstructure similar to -26 but more porous. Evident W solubility, second phase in metal globs apparently W.

Table I (Cont'd)

Run #	Material (Wt %)	Remarks (°C)
W-Mp-76-28	21.5 Al_2O_3 48.5 Cr_2O_3 20 Cr 10 Fe	Repeat of -19 in tungsten, melted ~1790°, very find metal dispersions.
W-Mp-76-29	21.5 Al_2O_3 48.5 Cr_2O_3 10 Cr 20 Fe	Repeat of -20 in tungsten, melted ~1860°, more dense than -28, very fine eutectic, whiskers not crystallographic, excess metal, some primary oxide (Al_2O_3?), slight reaction with crucible.
W-Mp-76-30	21 Cr_2O_3 49 Fe_2O_3 6 Cr 24 Fe	Repeat of -21 in W, melted ~1865°. Good disperse metal-metal oxide throughout. 2-phase metal reaction layer at W wall, internal globs appear to be single phase.

Table II

Directional Solidifications in Tungsten Tubes

Run #	Material (Wt %)	Remarks
W-DS-75-12	33.57 Al_2O_3 49.46 Cr_2O_3 16.97 WO_3	Used 3% Polystyrene binder, 4" long CVD tungsten tube, 3 susceptors. Plug down 1900° Obs., considerable smoking and melt through.
W-DS-75-13	33.57 Al_2O_3 49.46 Cr_2O_3 16.97 WO_3	Repeat of previous run, used vibrator, plug down 1950°. Lower 1/4" not melted, ingot ~7/8" long with large air space above.
W-DS-75-14	33.57 Al_2O_3 49.46 Cr_2O_3 16.97 WO_3	Extended length of crucible (tube) to ~7", vibrator at 100 cps. Obtained slug about 3 1/2" long but with many voids.
W-DS-75-15	33.57 Al_2O_3 49.46 Cr_2O_3 16.97 WO_3	Remelt of previous run, outgassing and smoking. Weight on charge rod to indicate melting down at 1980°C, traverse 2 cpm. Crucible broke in half about 2 1/8" from bottom.
W-DS-75-16	54.5 Cr_2O_3 24.5 Al_2O_3 21.0 Cr	7 3/4" long charge, vibrator 100 cps, plug down 1738°, traverse 3.2 cm/hr. Plug never came down. Tear in crucible ~3/16" long. Slight amount of melt came through. Ingot 1 3/4" long, came free of crucible, less voids, some banding.
W-DS-75-17	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Plug down at 1800°C, Traverse failed to work.

Table II (Cont'd)

Run #	Materials (Wt %)	Remarks
W-DS-75-18	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Lower heater at 26 amps, plug down with help of vibrator at 1680°, some distortion of W observed, 1 cm/hr. Upper section of crucible sealed by ingot, gas pressure broke crucible. Ingot 1 1/2" long, few voids, longitudinal cracks in some area, good alignment, non cellular-looks good for further works.
W-DS-75-19	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Lower heater at 26 amps, vibrator 1000 cps, plug down slowly at 1630°, cooled with lower heater on automatic. Slight indication of leakage at seam between crucibles, good slug 2" long, metal dendrites and some internal globs of metal.
W-DS-75-20	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	At 1900°C; 2, 1.6, 1.0 and 0.6 cm/hr. Melt came through crucible. Bottom 1" long ingot, 1 3/8" air pocket then an irregular 1/4" thick slug sealed crucible.
W-DS-75-21	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	4 cm/hr, then 2.3 and 2.1 cm/hr. Crucibles clean and intact except lower crucible was bowed. Ingot 2 1/4" long, low 1" irregular in shape. MOR samples made. Samples need more metal.
W-DS-75-22	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Run to investigate use of tantalum tube with an internal CVD coating of W. Crucible hung up, wetted heater.
W-DS-75-23	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	1.5 cm/hr, vibrator 1.5 amps, 500 cps. Material came out through crucible, crucible extensively wetted. Ingot 3/4" long produced.

Table II (Cont'd)

Run #	Materials (Wt %)	Remarks
W-DS-75-24	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Plug held up because of melting adjacent to Grafoil disks, all material below melted o.k.
W-DS-75-25	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Crucible bowed as it left heater, two crucibles (tubes) came out intact but lower crucible leaked in two areas. Ingot 1 7/8", one pore.
W-DS-75-26	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Run made without Grafoil above susceptor. Plug started down at 1740°, melt came through crucible. Did uneven tungsten coating cause a weak spot?
W-DS-75-27	54.4 Cr_2O_3 22.6 Al_2O_3 23 Cr	Replaced Grafoil above susceptor, used two tungsten tubes, ingot 2" long, one air pocket.
W-DS-75-28	38.5 Cr_2O_3 38.5 Al_2O_3 23 Cr	4" tungsten-coated Ta tube. Material broke through 1 1/4" from top but no visible leaking, Ingot 1 3/8" long, contained some voids.

Table II (Cont'd)

Run #	Material (Wt %)	Remarks
W-DS-75-29	23 Cr 38.5 Cr_2O_3 38.5 Al_2O_3	Used 7" long Ta tube with internal [illegible] Tungsten coating. Crucible removed intact, 2" long section free of voids; 0.5 cm/hr, Temp. 1760° C Obs.
W-DS-75-30	46 Cr 27 Al_2O_3 27 Cr_2O_3	7" long Ta tube, Tungsten I. D., 0.5 cm/hr. Crucible failed, salvaged 1" long slug, four distinct areas with different metal compositions.
W-DS-75-31	43 Cr 28.5 Al_2O_3 28.5 Cr_2O_3	Ta - W crucible as above, badly burned through. Indicated temp. 1750° C.
W-DS-75-32	40 Cr 30 Al_2O_3 30 Cr_2O_3	Tungsten tube, 0.010" walls, plasma sprayed. Big section of crucible dissolved or "burned" away.
W-DS-75-33	23 Cr 22.6 Al_2O_3 54.4 Cr_2O_3	Repeat of run -21 . W tube CVD at UTRC, Wt 11 gms, greatest wall thickness .012". Traversed 0.8 cm/hr, crucible removed intact. $2\frac{1}{2}$" long ingot with two large air pockets.
W-DS-75-34	23 Cr 22.6 Al_2O_3 54.4 Cr_2O_3	Repeat of run -21. Crucible 20 gms (UTRC). Extensive damage to crucible, $\frac{1}{2}$" long ingot produced.
W-DS-75-35	23 Cr 22.6 Al_2O_3 54.4 Cr_2O_3	UTRC crucible, without lower heater, crucible failed, shut down.

Table II (Cont'd)

Run #	Material (Wt %)	Remarks
W-DS-75-36	23 Cr 22.6 Al_2O_3 54.4 Cr_2O_3	UTRC-CVD-W crucible, new plug 98 gms., 0.6 cm/hr. Crucible O.K., plug dropped only 2".
W-DS-75-37	23 Cr 22.6 Al_2O_3 54.4 Cr_2O_3	UTRC-CVD-W crucible, new plug 57 gms. Charge was ingot from run -36., 0.6 cm/hr, 3/4" long ingot, rest of ingot lost through crucible. Remelting did not remove fine scale porosity.
W-DS-75-38	23 Cr 20.78 Al_2O_3 20.78 Cr_2O_3 30.13 ZrO_2 5.32 Y_2O_3	UTRC-CVD crucible, 25 gm, 7" long, 0.6 cm/hr. Ingot 1½" long, plug sealed crucible 1½" from top, pressure built up and broke crucible. Cutting heater length from 1 7/8" to 1 3/16".
W-DS-75-39	23 Cr 20.78 Al_2O_3 20.78 Cr_2O_3 30.13 ZrO_2 5.32 Y_2O_3	0.6 cm/hr, crucible came out intact, only damage a pin hole ~ 3½" from bottom, 2" long.
W-DS-75-40	23 Cr 20.78 Al_2O_3 20.78 Cr_2O_3 30.13 ZrO_2 5.32 Y_2O_3	UTRC-CVD-W crucible again, 16 gms., run 0.6 cm/hr. Melt broke through; ingot ~ ½" long

Table II (Cont'd)

Run #	Material (Wt %)	Remarks
W-DS-75-41	23 Cr 20.78 Al_2O_3 20.78 Cr_2O_3 30.13 ZrO_2 5.32 Y_2O_3	1.1 cm/hr, crucible ruptured and deformed in two places. Conclusion: the CVD process must be made in one continuous operation.
W-DS-75-42	20 Cr 40 Al_2O_3 40 Cr_2O_3	Most of melt lost through break in CVD crucible.
W-DS-75-43	24 Cr 28 ZrO_2 4.6 Y_2O_3 21.7 Al_2O_3 21.7 Cr_2O_3	Run 1 cm/hr, break-out 2" from bottom, air pocket and melt sealed at top caused failure. Ingot 1½" long. May not have been hot enough, lower part of ingot porous.
W-DS-75-44	24 Cr 28 ZrO_2 4.6 Y_2O_3 21.7 Al_2O_3 21.7 Cr_2O_3	Crucible ruptured about 3" from bottom, no leakage, 2" long ingot prepared.
W-DS-75-45	24 Cr 28 ZrO_2 4.6 Y_2O_3 21.7 Al_2O_3 21.7 Cr_2O_3	0.58 cm/hr, crucibles intact but slight leakage. Two ingots separated by air pocket, 1 1/8" and 1" long. Good structures but cellular.
W-DS-75-46	20 Cr 40 Al_2O_3 40 Cr_2O_3	Plasma sprayed W tube, 10 gms., ~ .010" walls. 1.6 cm/hr. Crucible badly distorted but in one piece.

Table II (Cont'd)

Run #	Material (Wt %)	Remarks
W-DC-75-47	23 Cr 25 ZrO_2 4.Y_2O_3 23.5 Al_2O_3 23.5 Cr_2O_3	Crucible failed, materials fused to susceptor, total loss of melt. Failure due to improper use of stretch-squeeze mechanism.
W-DC-75-48	23 Cr 25 ZrO_2 4.Y_2O_3 23.5 Al_2O_3 23.5 Cr_2O_3	Plasma sprayed W crucible, new susceptor, 2.2 and 1 cm/hr. Crucible has no strength, distorted and contacted susceptor.
W-DC-75-49	23 Cr 25 ZrO_2 4.Y_2O_3 23.5 Al_2O_3 23.5 Cr_2O_3	UTRC-CVD-W crucible, .007 to .010" walls. Crucible in excellent shape, ingot 1" followed by small air pocket and another ½" of ingot.
W-DC-75-50	23 Cr 25 ZrO_2 4.Y_2O_3 23.5 Al_2O_3 23.5 Cr_2O_3	UTRC-CVD-W crucible, .006" to .012" walls. Ingot 1 3/4" long, some material lost through crucible. Ingot grown for short length off sapphire seed parallel to $[02\bar{2}4]$.
W-DC-75-51	23 Cr 54.4 Cr_2O_3 22.6 Al_2O_3	UTRC-CVD-W crucible, 10 gms., .52 and 2 cm/hr. Started well but slightly metal rich, bands present, areas both oxide or metal rich, metal precipitate between whiskers and apparent fine scale porosity.

Table II (Cont'd)

Run #	Material (Wt %)	Remarks
W-DB-75-52	22 Al_2O_3 53 Cr_2O_3 25 Cr	UTRC-CVD-W crucible, 15 gms, .5 cm and then 2 cm/hr. Melt came out of tube at bottom, bridging to susceptor, air pocket formed, most of material did not melt.
W-DB-75-53	23 Al_2O_3 56 Cr_2O_3 21 Cr	UTRC-CVD-W crucible (tube). Wt started down 1900°C, 1 1/2" ingot, oriented but cellular. All parts with regions of apparent fine porosity.
W-DB-75-54	23 Al_2O_3 56 Cr_2O_3 21 Cr	0.5 and then 2 cm/hr, 2040°C. Slight leak in crucible about 2 1/2" from bottom, 2" long ingot, oriented sections throughout, but much structure random, metal poor. Still some large scale, slightly oriented porosity, perhaps fine scale porosity also.
W-DB-75-55	22.013 Al_2O_3 53.987 Cr_2O_3 25.0 Cr	UTRC-CVD-W crucible, very close to eutectic, perhaps slightly more metal needed, fine "porosity" effect probably worsened by metal ppt 2 1/8" ingot, 3/8" air pocket.
W-DB-75-56	22.6 Al_2O_3 54.4 Cr_2O_3 23 Cr	UTRC-CVD-W crucible, 1880-1890°C, 0.5 and 2 cm/hr, 2" long ingot, no leakage, looks on eutectic, slightly metal rich near end, alignment but not uniform, fine "porosity".
W-DB-75-57	22 Al_2O_3 54 Cr_2O_3 24 Cr	UTRC-CVD-W crucible, 23 gm, 2" ingot, dense, 1st half done in vacuum, last half 1 atm. argon. Sample poor in metal, 1/8-1/4" eutectic, fine scale "porosity" missing in eutectic but obvious voids fairly extensive.

Table II (Cont'd)

Run #	Material (Wt %)	Remarks
W-DS-75-58	42 Al_2O_3 42 Cr_2O_3 16 Cr	UTRC-CVD-W crucible, 23 gm, 1 cm/hr, ingot 1 3/4", some metal break through. Some very good areas but porosity still a problem. Metal poor.
W-DS-75-59	44 Al_2O_3 44 Cr_2O_3 16 Cr	UTRC-CVD-W crucible, 1st 27 min in vacuum by mistake at 1640°C, sample quite metal poor but still grain boundary porosity, ingot 1 5/8".
W-DS-75-60	43 Al_2O_3 43 Cr_2O_3 14 Cr	UTRC-CVD-W crucible, sample length 1 7/8".
W-DS-75-61	43 Al_2O_3 43 Cr_2O_3 14 Cr	UTRC-CVD-W crucible, used ingot from W-DS-75-60 as charge, D.S. 1 cm/hr, some material came through crucible, ingot 1 1/4" long, still shows porosity.
W-DS-75-62	42 Al_2O_3 42 Cr_2O_3 16 Cr	UTRC-CVD-W crucible, tried to seed with Al_2O_3 crystal, 0.5 and 1 cm/hr, 5/8" long ingot, not hot enough to seed or fuse to sapphire.
W-DS-75-63	42 Al_2O_3 42 Cr_2O_3 16 Cr	UTRC-CVD-W crucible, ingot 3/4" long, some leakage. Some extensive areas of good eutectic, transparent oxide in center of ingot.
W-DS-75-64	42 Al_2O_3 42 Cr_2O_3 16 Cr	UTRC-CVD-W crucible, sapphire seed and W funnel, ingot on 1/8" long, break in W crucible; it appeared funnel was reacting.

Table II (Cont'd)

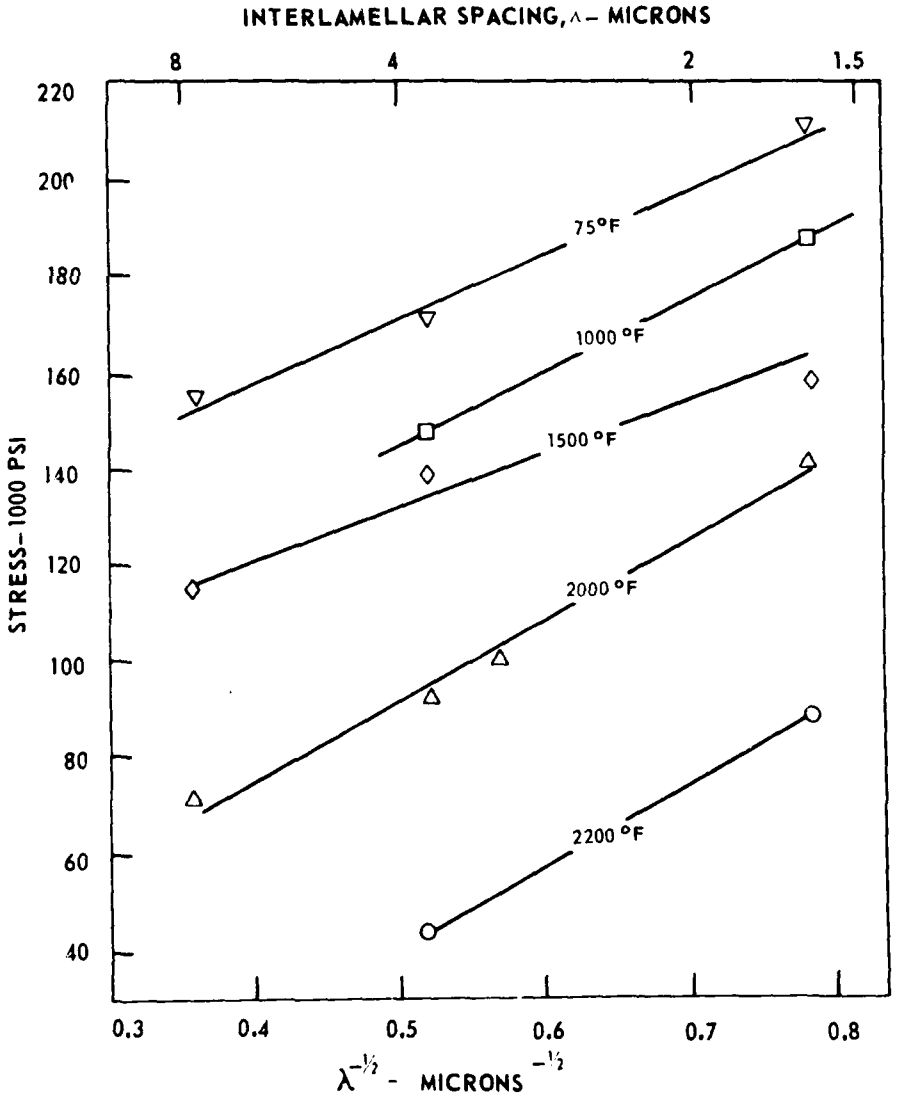
Run #	Material (Wt %)	Remarks
[illegible]	23.6 Al_2O_3 54.4 Cr_2O_3 23 Cr	Most of material lost through crucible, some bridged to heater insulation, ingot ~1/2" long with center shrink hole. Microstructure shows some regular porosity, not associated with best eutectic areas. Growth not well oriented, looks like Al_2O_3-Cr_2O_3 eutectic.
W-DS-76-18	23.6 Al_2O_3 54.4 Cr_2O_3 23 Cr	1.6 cm/hr, good slug 1 11/16" long, crucible in good condition, .0135" dia holes put in W for gases to escape; temperature 1822°, microstructure cellular, alignment imperfect with porosity.
W-DS-76-19	23.6 Al_2O_3 54.4 Cr_2O_3 23 Cr	1.5 cm/hr, reached 1830°C, melt came through crucible and bridged to furnace.
W-DS-76-20	23.6 Al_2O_3 54.4 Cr_2O_3 23 Cr	Reached 1790°C, crucible leaked and bridged, 3/16" ingot.
W-DS-76-21	23 Al_2O_3 52 Cr_2O_3 16.7 Fe 8.3 Cr	4.2 cm/hr, reached 1920°, crucible collapsed about 4" from bottom, 3/4" ingot at bottom, 1/2" at collapsed area. Went too high in temperature. In this time frame - difficulty in making single run CVD tubes of good quality and thickness.
W-DS-76-22	22.5 Cr_2O_3 52.5 Fe_2O_3 20.0 Fe 5.0 Cr	Reached 1715°, material went through crucible, bridged to furnace. Metal insoluble in ceramic.

Table II (Cont'd)

Run #	Material (Wt %)	Remarks
W-DS-76-23	68.2 Cr_2O_3 11.8 Fe_2O_3 8.8 Fe 11.2 Cr	Reached 1740°, crucible deformed, charge did not melt.
W-DS-76-24	68.2 Cr_2O_3 11.8 Fe_2O_3 8.8 Fe 11.2 Cr	Remelt of run -23, noted cracks at base of crucible prior to run. Melt came through crucible and bridged. Ingot porous, metal rich.
W-DS-76-25	68.2 Cr_2O_3 11.8 Fe_2O_3 8.8 Fe 11.2 Cr	Reached 2053°, 1.5 cm/hr, 3/4" long slug + 1/2" long slug above gas pocket; only slight crucible attack. Good metal solubility but no D.S. ceramic microstructure, some porosity, some globs of metal (2-phase).
W-DS-76-26	23.0 Al_2O_3 52.0 Cr_2O_3 16.7 Fe 8.3 Cr	Sample broke out of crucible but other melted sections separated cleanly from tungsten. Good solubility metal may have separated in solid state process - in other areas, larger whisker-like metal phase but not regular.

TENSILE STRENGTH OF Ni_3Al–Ni_3Cb EUTECTIC AS A FUNCTION OF TEMPERATURE AND (INTERLAMELLAR SPACING)$^{-1/2}$

(REF. 1)



NON–PROPAGATING CRACK GENERATED IN FATIGUE SPECIMEN OF Ni_3Al–Ni_3Nb TESTED AT ± 135 KSI

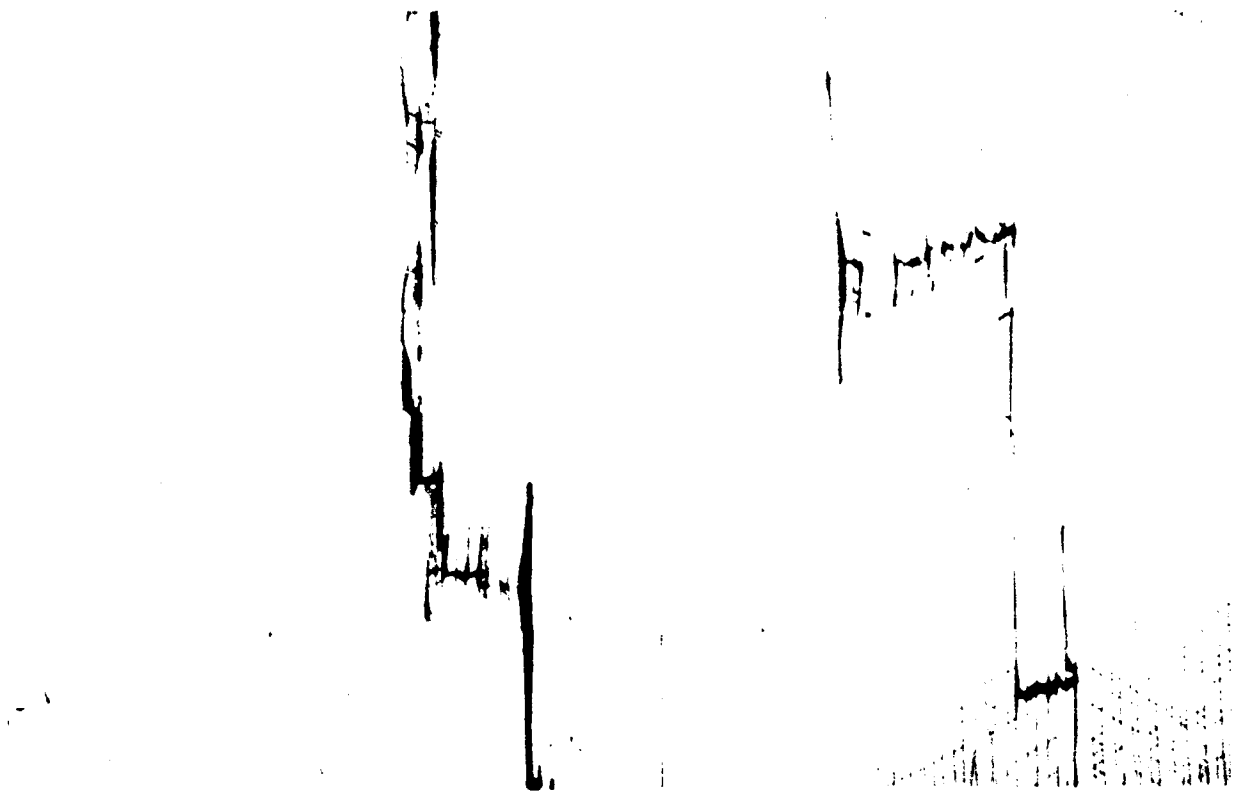
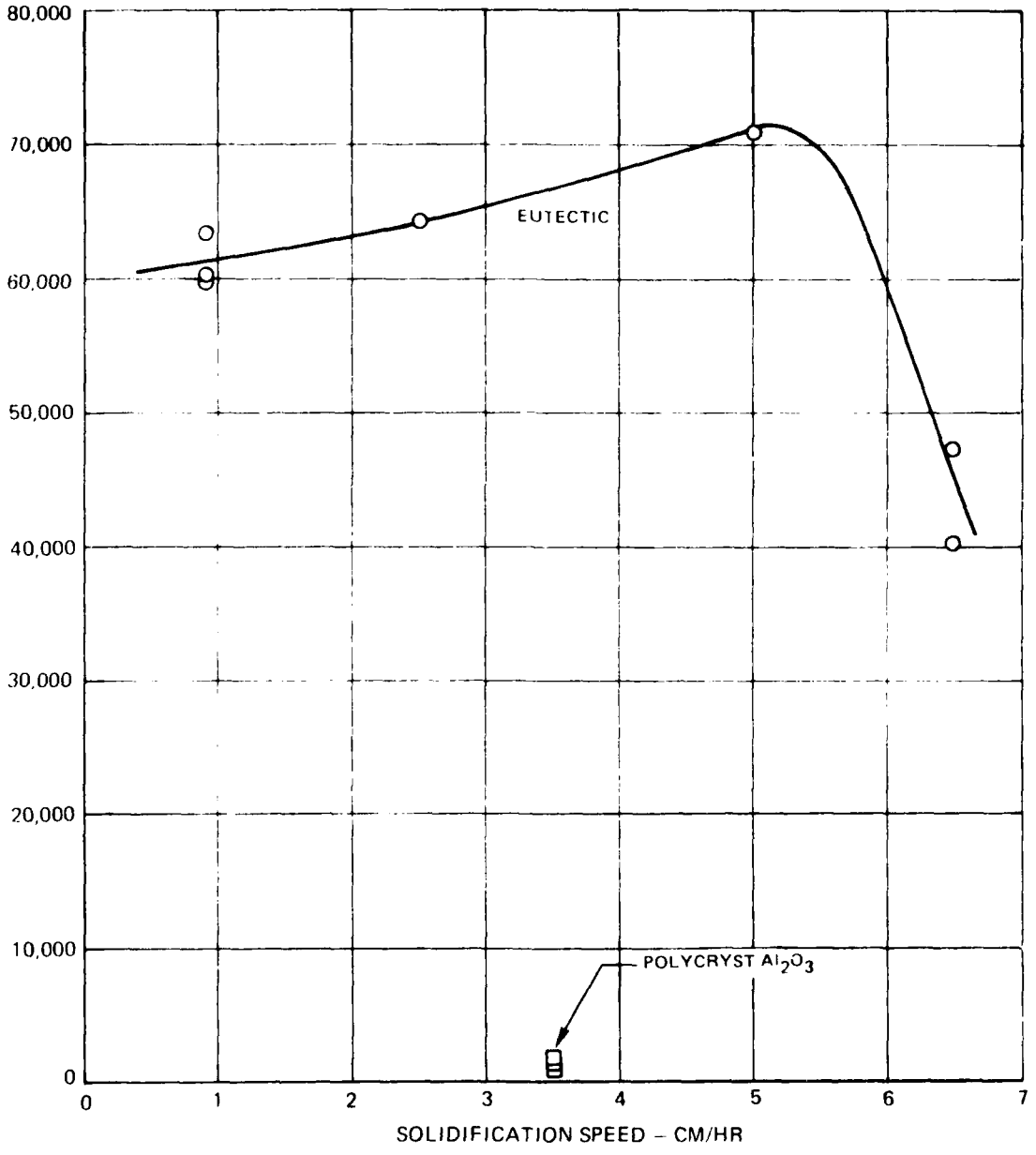


FIG. 2

STRENGTH OF Al_2O_3–ZrO_2 (Y_2O_3) EUTECTIC AT 1575°C COMPARED WITH POLYCRYST. Al_2O_3



WORK–TO–FRACTURE OF $CaO \cdot ZrO_2$–ZrO_2 EUTECTIC DIRECTIONALLY SOLIDIFIED AT 10 CM/HR AND POLYCRYST Al_2O_3 VERSUS TEMPERATURE

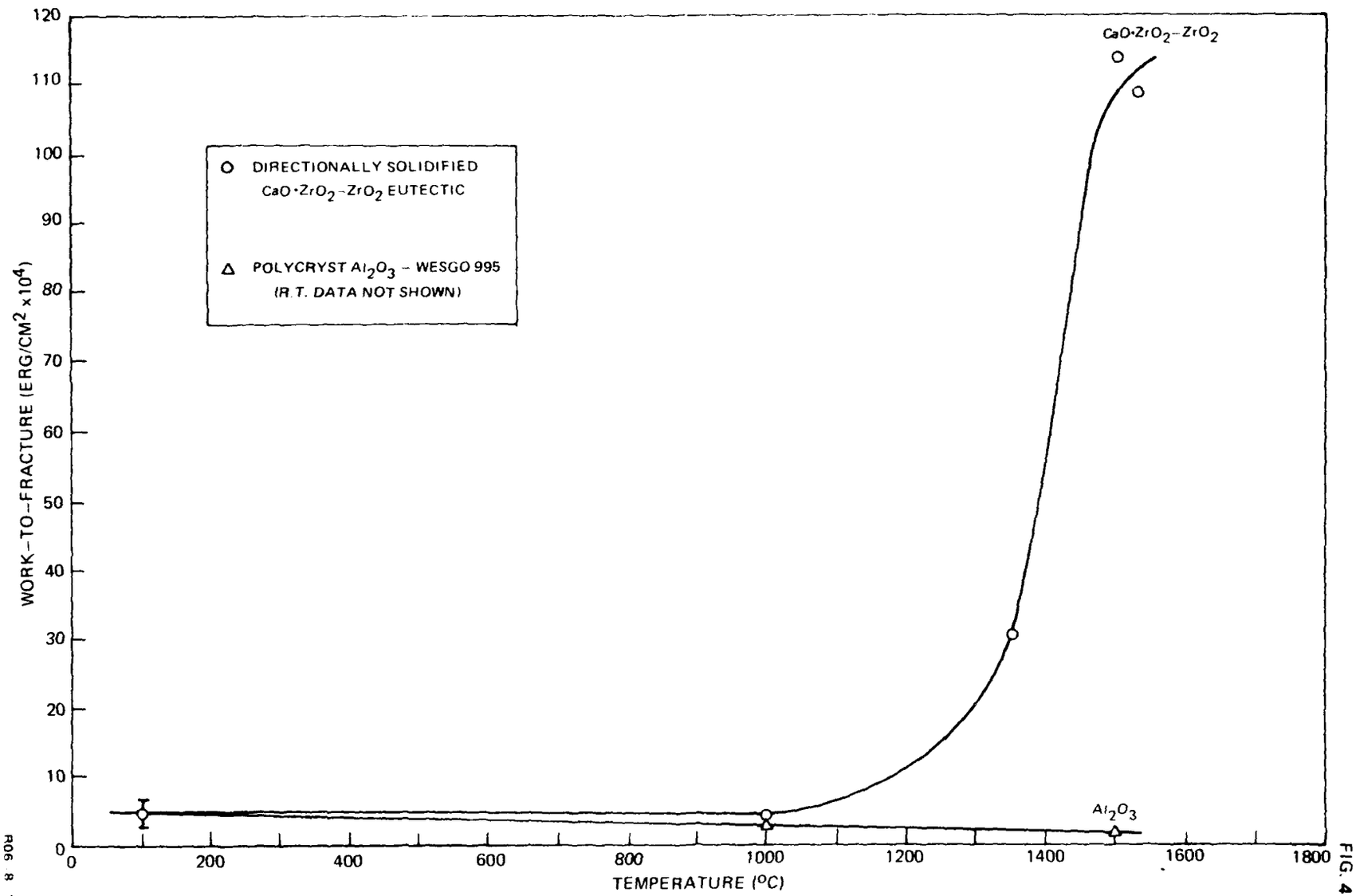
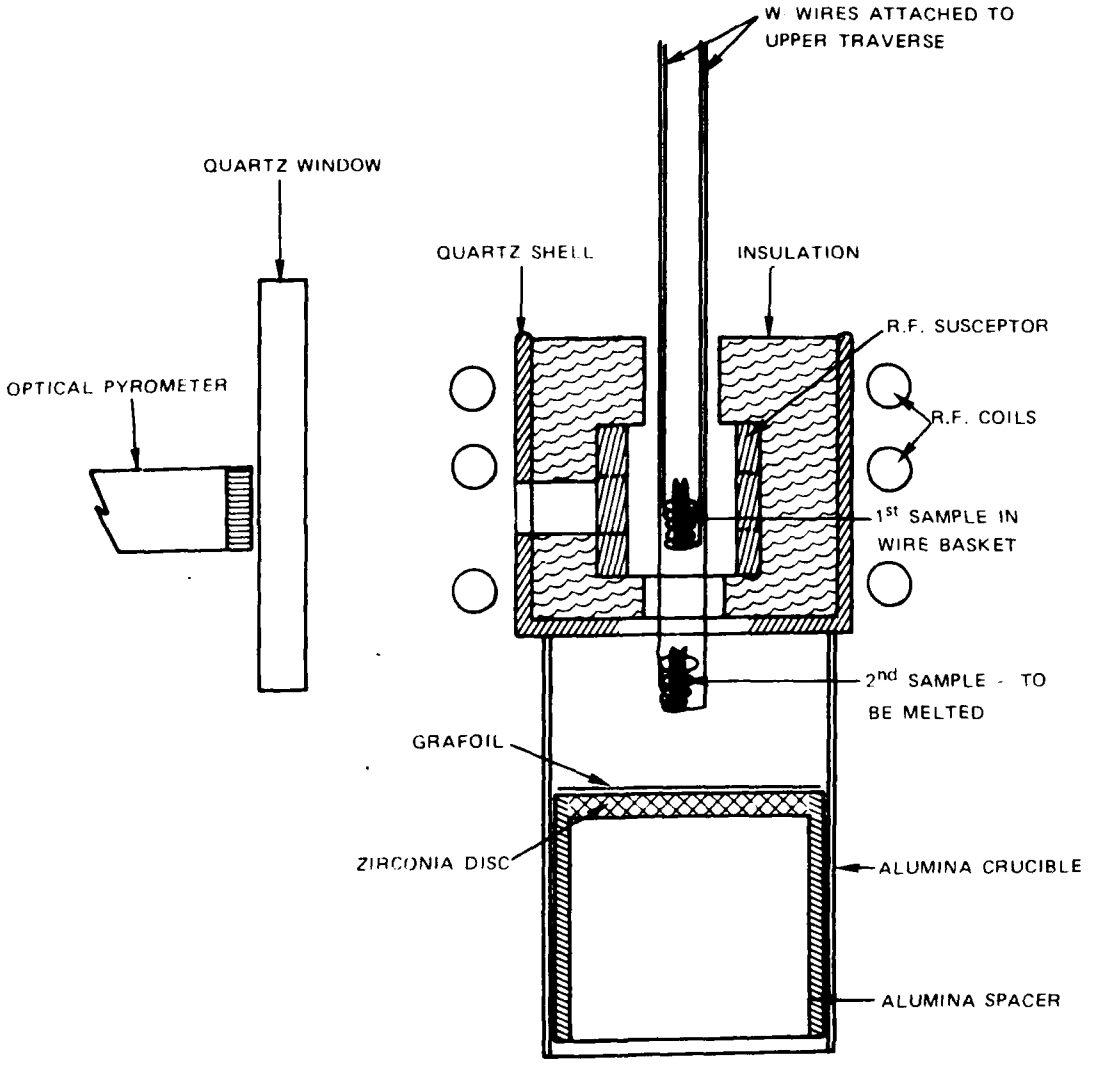


FIG. 4

R06 8 1

SCHEMATIC OF TUNGSTEN WIRE–BASKET MELTING POINT FURNACE



MELTING DATA FOR Cr–(0.7 Cr_2O_3, 0.3 Al_2O_3) USING TUNGSTEN BASKET IN ARGON WITH CARBON HEATER

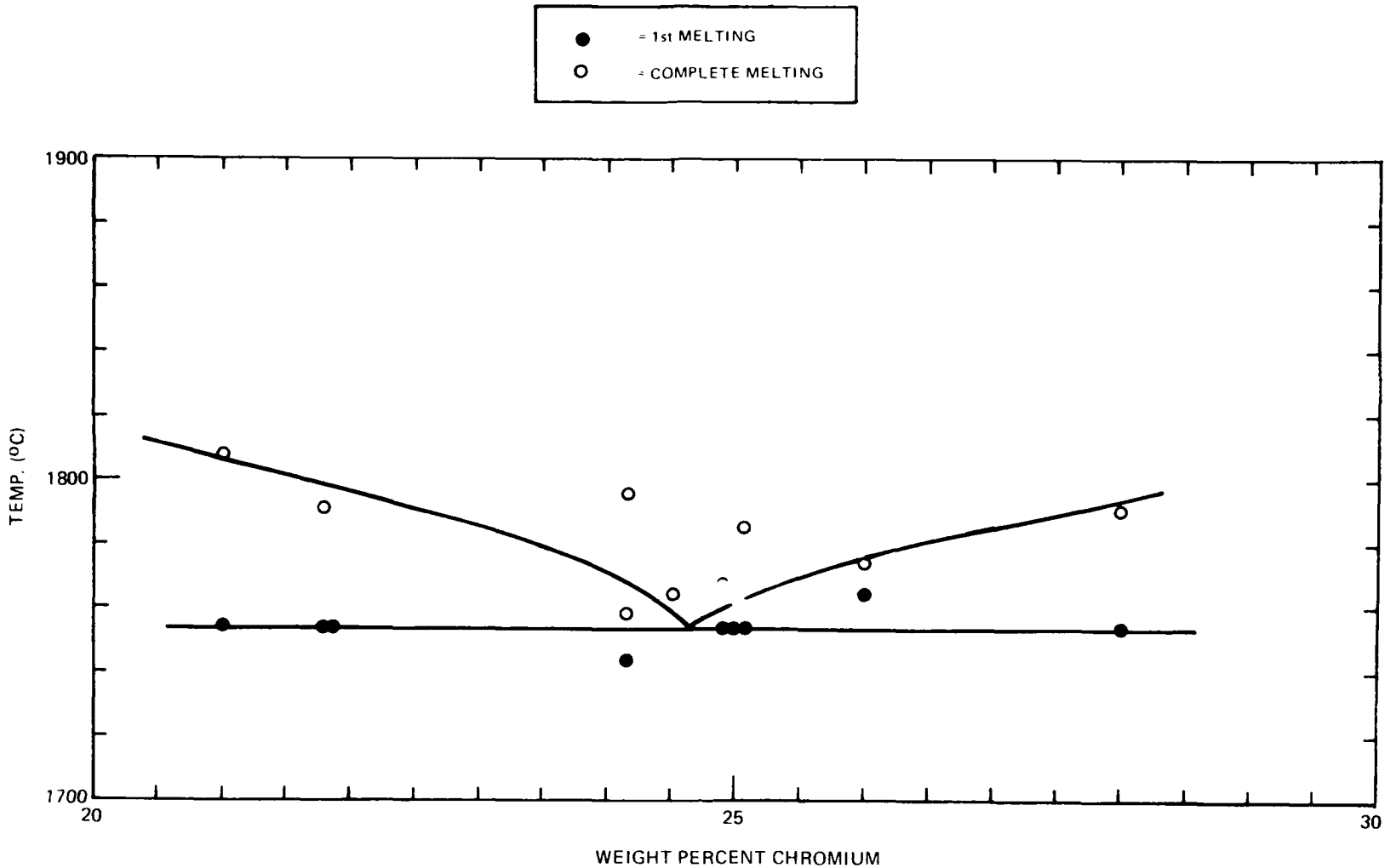
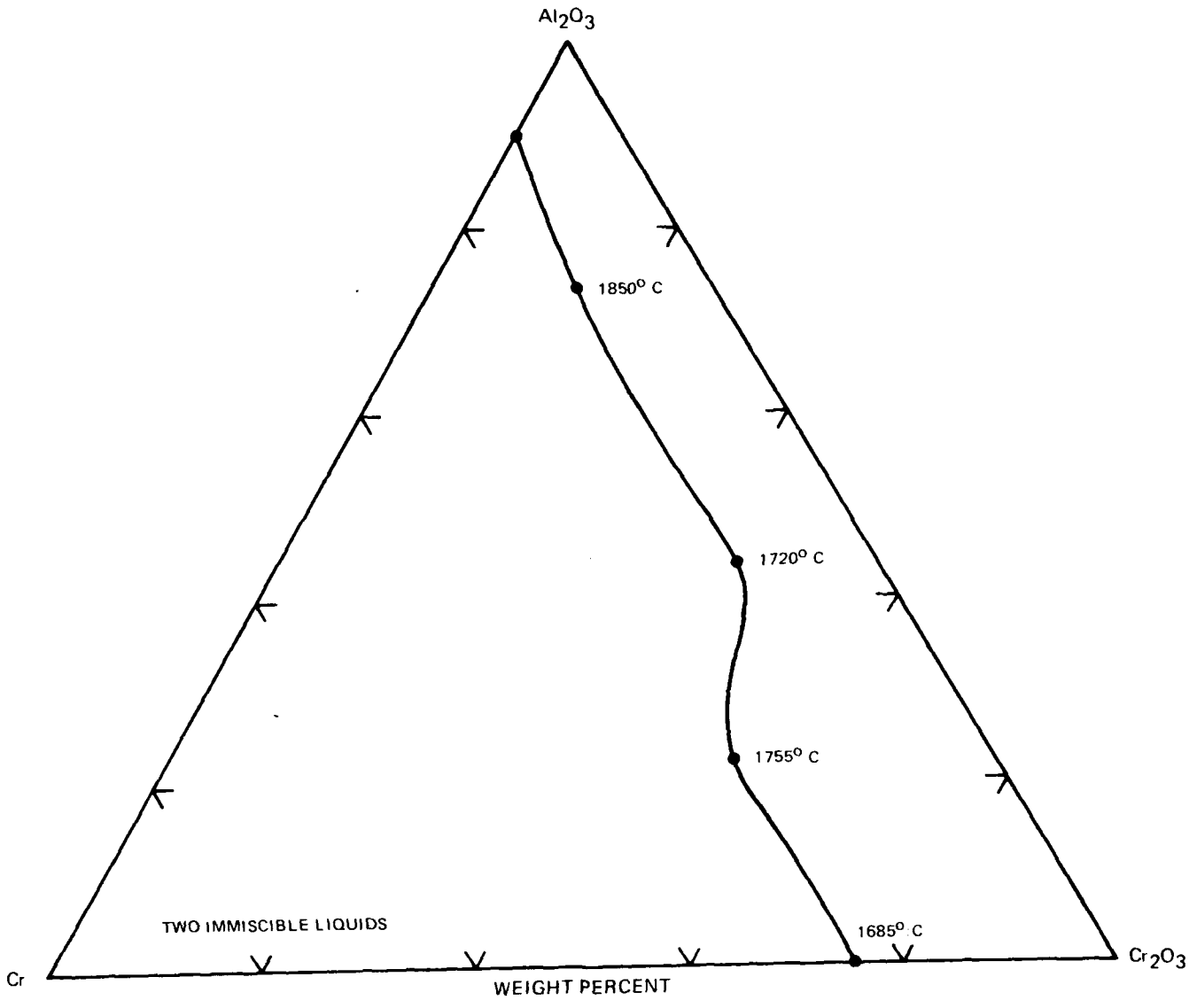


FIG. 6

76 06 111 4

BINARY EUTECTIC TROUGH IN $Cr-Al_2O_3-Cr_2O_3$ TERNARY IN ARGON USING CARBON HEATER



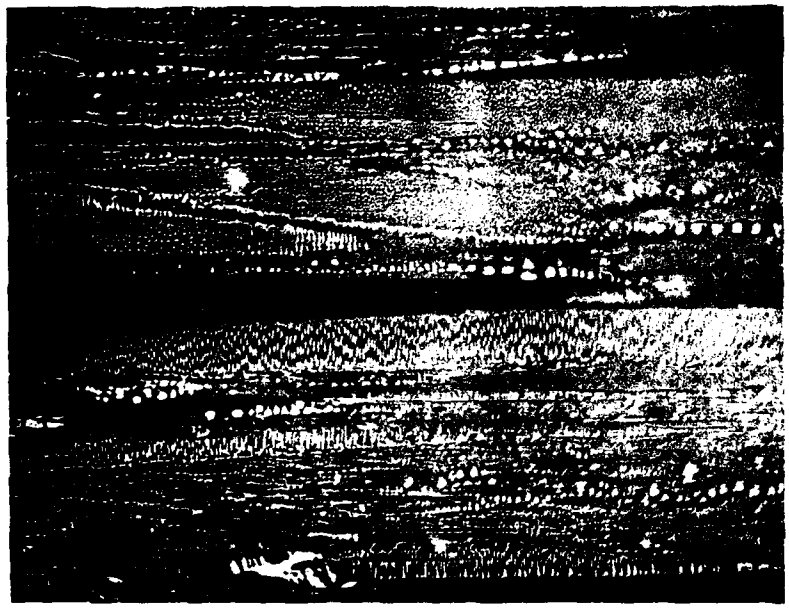
76 06-111 5

FIG. 7

FIG. 8

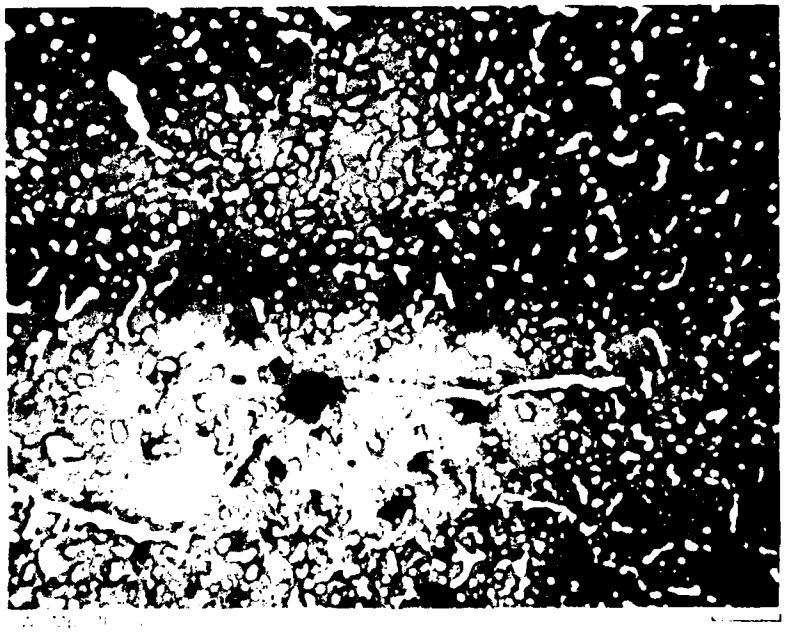
MICROSTRUCTURE FORMED BY MELTING MULLITE–ZIRCON EUTECTIC COMPOSITION

SOLIDIFICATION ⟶



25μ

MICROSTRUCTURES FORMED BY MELTING 21 w/o Cr_2O_3, 49 Fe_2O_3, 6 Cr AND 24 Fe



FURNACE FOR DIRECTIONAL SOLIDIFICATION OF CERAMIC-MELT METAL EUTECTICS

(VERTICAL CENTER SECTION VIEW)

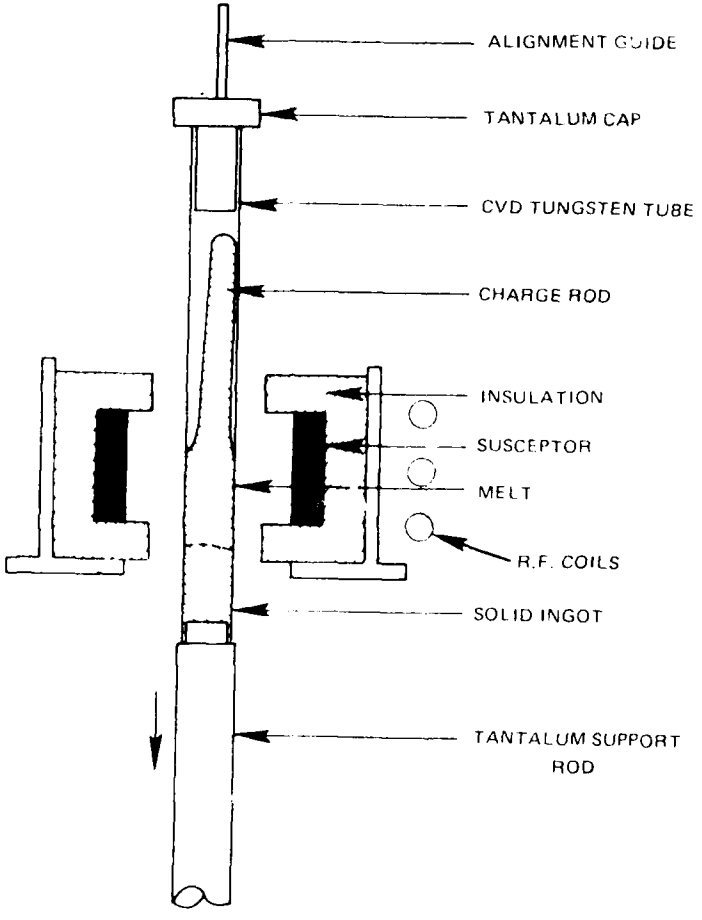


FIG. 10

FIG 11

TRANSVERSE MICROSTRUCTURE OF BINARY EUTECTIC

[illegible] Cr_2O_3 - Al_2O_3 DIRECTIONALLY SOLIDIFIED 1.6 CM/HR

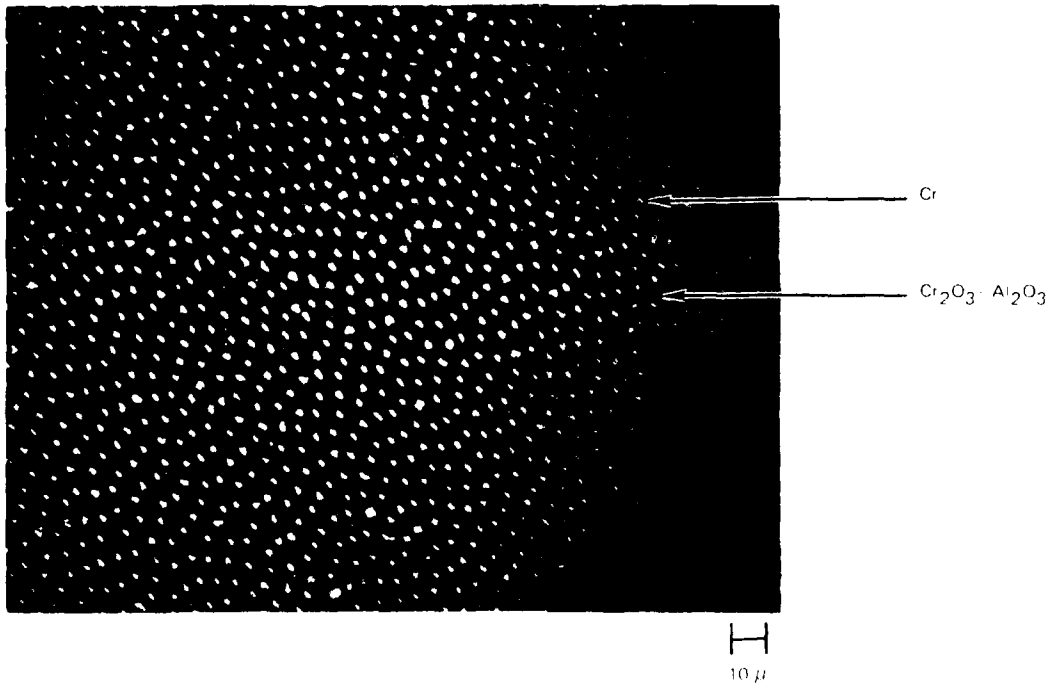


FIG. 12

LONGITUDINAL MICROSTRUCTURE OF TERNARY EUTECTIC
24 w/o Cr, 28 w/o ZrO_2, 4.6 w/o Y_2O_3, 21.7 w/o Cr_2O_3, 21.7 w/o
Al_2O_3 DIRECTIONALLY SOLIDIFIED AT 0.5 CM/HR

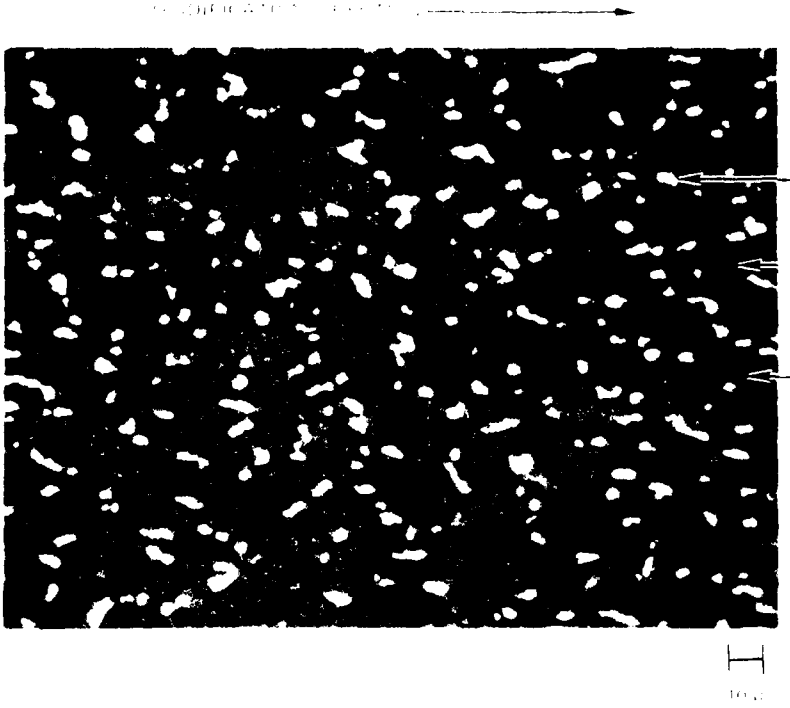
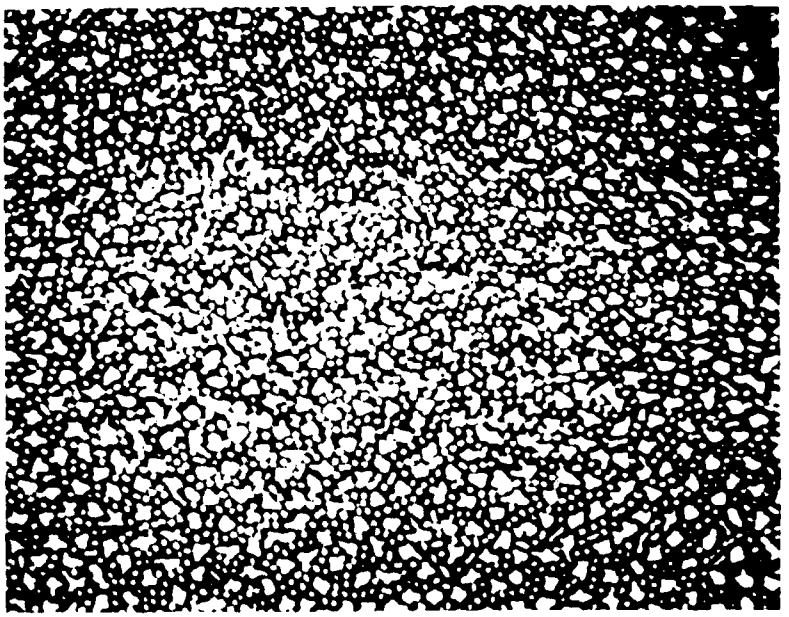


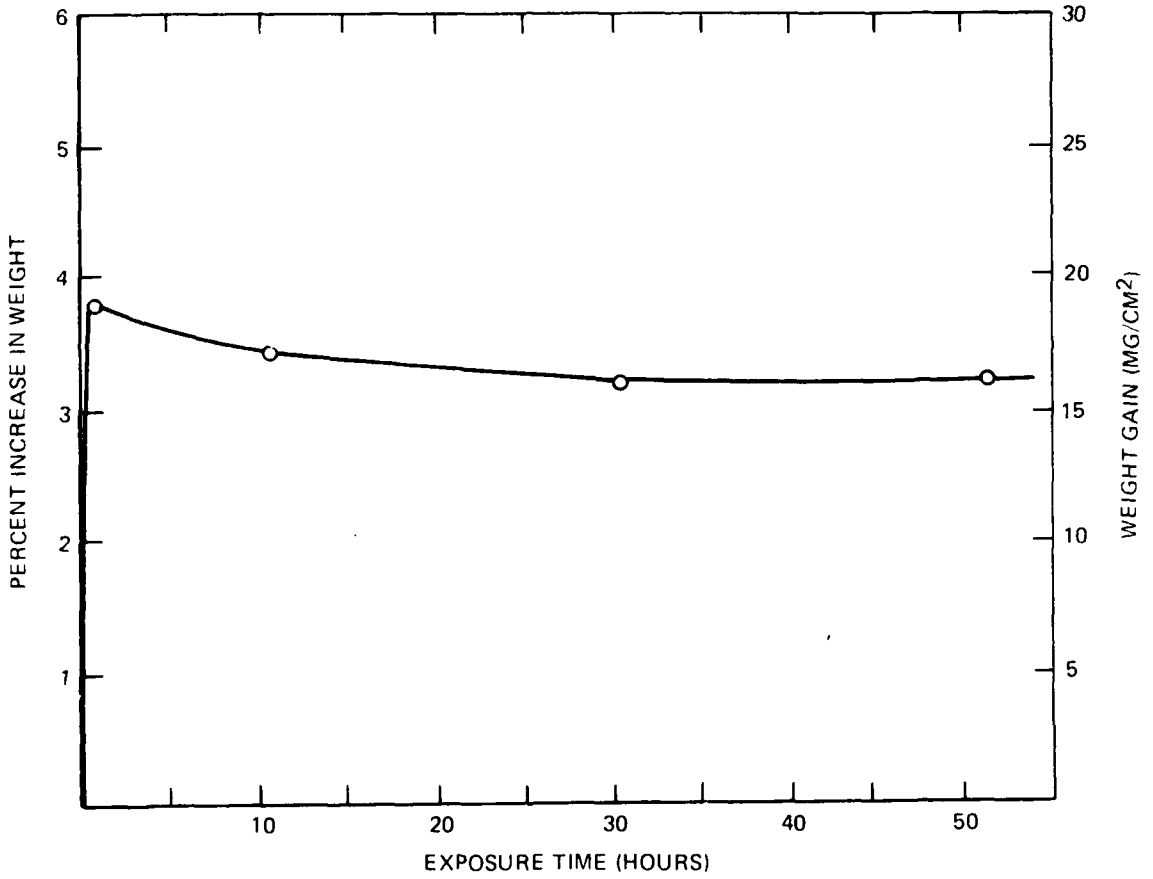
FIG. 13

TRANSVERSE MICROSTRUCTURE OF 24 w/o Cr, 23.5 Al_2O_3, 23.5 Cr_2O_3, 25 ZrO_2 AND 4Y_2O_3 EUTECTIC GROWN PARALLEL TO THE |0224| OF A SAPPHIRE SEED



WEIGHT GAIN OF DIRECTIONALLY SOLIDIFIED TERNARY EUTECTIC

24 w/o Cr, 28 w/o ZrO_2, 4.6 w/o Y_2O_3, 21.7 Cr_2O_3, 21.7 w/o Al_2O_3 IN AIR AT 1425°C



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