INVESTIGATION OF BONDING IN OXIDE-FIBER (WHISKER) REINFORCED METALS

By

Willard H. Sutton

November 1962

Space Sciences Laboratory
Missile & Space Division
General Electric Company
Philadelphia, Pennsylvania

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ABSTRACT

One of the critical problems in achieving high strength composites by reinforcing metals with oriented single-crystal fibers (whiskers) is the strength of the interfacial bond between the two phases. The purpose of this program, therefore, is to investigate the factors affecting interfacial bonding so that appropriate bonding techniques can be specified for the fabrication of high strength composites. This report describes the problem areas and the experimental procedures to be used in the investigation. In order to study and evaluate the effects of various parameters affecting interfacial energies and bond strength, a sessile drop apparatus is being constructed. Future plans utilizing this equipmnet in conjunction with actual measurements of bond strengths are discussed.

I. INTRODUCTION

The reinforcement of metals and alloys with ultra high-strength filaments (whiskers) offers an important new approach for greatly strengthening metals and alloys over wide temperature ranges. On a strength-to-weight basis, this reinforcement could result in materials with superior structural performance for applications such as rocket cases, hypervelocity cruise vehicles, and armament.

It has been demonstrated experimentally that whisker reinforcement is feasible. For example, the reinforcement of silver with 24% α-Al_2O_3 whiskers resulted in a material which was six times stronger than the pure metal. (Ref. 1). These results are encouraging and indicate a promising future for whisker-reinforced metals. Before these composites become practical structural materials, however, several problems must be solved.

One of the most critical problems is associated with the strength of the bond at the fiber*-matrix interface. This bond must be very strong if high strength composites are to result, since the matrix must effectively transfer the load to the fibers via shear stresses at the interface. In the case of fibers oriented parallel to an applied load, these stresses are apt to peak to high values near the ends of the fibers. This fact was emphasized by Dow (Ref. 2) who performed a theoretical stress analysis of Al-α-Al_2O_3 (whisker) composites and concluded that the peak shear stresses near the whisker ends necessitated a strong, resilient bond at the metal-whisker interface.
An investigation** of silver - α-Al_2O_3 composites conducted in this laboratory has shown that special preparation of the whisker surfaces is needed to promote sufficient wetting and bonding to the metal matrix. It was found, for example, that metallizing the whiskers prior to infiltration of the silver matrix would serve this purpose; in every case where this treatment was not included, the whiskers would pull out of the matrix

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*The terms fiber and whisker (single crystal filament) are used interchangeably.

**Sponsored by the U.S. Navy, Bureau of Weapons, Contract NOw 60-0465d.

during tensile testing, and no significant improvement in strength was observed. On the other hand, when the Al_2O_3 whiskers were metallized, 'pullout' occurred less frequently, and notable strengthening was observed (Ref. 3). The effect of metallizing the whisker surfaces prior to fabrication is shown in Figure 1.

It is the purpose of the present investigation to study the fundamental factors affecting the wetting and bonding of α - Al_2O_3 whiskers and nickel. The prime objective is to develop high-strength bonds between the two phases, and ultimately to evaluate the bond strength by testing α - Al_2O_3 whisker-nickel composites. The initial bonding studies, which will be described in greater detail, will be based on sapphire (single crystal α - Al_2O_3)-nickel systems. This report includes the program plans and work accomplished during the first quarter from July 1 to September 30, 1962.

(a) Strong Silver-Al_2O_3 (Whisker) Composite. Whiskers Were Precoated (Metallized) (33X)

(b) Weak Silver-Al_2O_3 (Whisker) Composite Showing 'Pull-out' at the Fracture. Whiskers Were Not Metallized (70X)

Figure 1. Fractures of Silver-Al_2O_3 (Whisker) Wires Tested in Tension Showing the Effect of Metallizing Whiskers on Composite Strength.

II. METAL-OXIDE BONDING: NATURE OF THE PROBLEM

The (metallic) bonds associated with metals are generally not compatible with those associated with oxide systems, which are primarily ionic and covalent in nature. In addition, metals usually exhibit higher coefficients of thermal expansion, thereby further complicating the problem of adherence to an oxide material. This problem can be solved by providing an intermediate phase which serves as a 'bridge' between the metal and oxide. This 'bridge' alleviates the bonding, structural, and thermal expansion differences between the metal and the oxide. In some cases the 'bridge' may be simply an intermediate (sub or defect) oxide between the metal and its stable oxide (e.g., Fe-FeO-Fe_2O_3), or it may consist of a foreign phase (e.g., Al_2O_3-TiO-Ti-Metal).

Much of the information regarding the bonding of metals to oxide (ceramic) systems has been made available by the electronic-tube industry, where hermetic metal-glass and metal-ceramic seals are of prime concern. Since this information has a bearing on the bonding of metals to oxides, it will be included in this discussion. It should be emphasized, however, that a hermetic seal does not imply strong bonding. Hermetic seals frequently show a considerable degree of porosity in the metal-oxide interface (Ref. 4). Since the pores are not continuous, the seals are vacuum tight. On the other hand, porosity or poor contact between the metal-whisker interface cannot be tolerated if high strength composites are to be achieved.

In this program emphasis will be placed on developing chemical bonds between the metal and the oxide, since they are far stronger than either mechanical bonds (caused by friction and interlocking of roughened surfaces) or Van der Waal's bonds. The latter bonds, in particular, may have only about 1/100 the strength of chemical bonds.

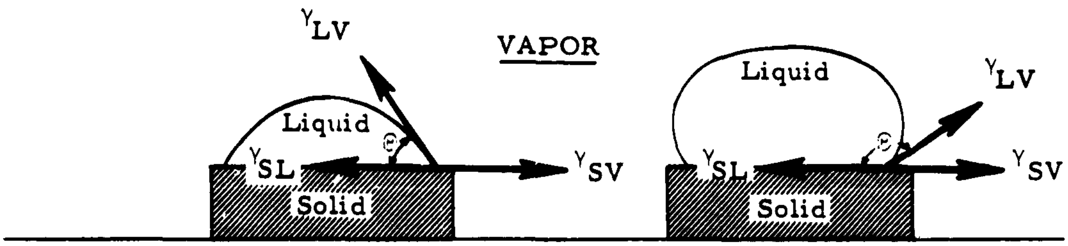
The chemical bonding of metals to oxides involves both wetting and adhesion. These factors are dependent on the relative surface energies of the phases involved and on the nature of the intermediate phase between the metal and oxide. Wetting implies an immediate attraction between a liquid and a solid once contact is made. Actually, wetting is a phenomena which is determined by the surface energies of all phases (solid, liquid, vapor) present. One technique which clearly demonstrates the equilibrium relationships between the solid-liquid (γ_{SL}), liquid-vapor (γ_{LV}), and solid-vapor (γ_{SV}) interfacial energies is a sessile drop experiment. The equilibrium shape of a liquid drop on a solid surface is dependent on the magnitude of the three surface energies and on the drop density for any given temperature. These relationships are schematically and mathematically shown in Figure 2, and are referred to in the ensuing discussion.

The sessile drop (in this case, a liquid metal drop on a solid oxide surface) provides a direct indication of the degree of 'wetting'; if the contact angle θ is greater than 90°, a condition of nonwetting is said to exist. However, if θ is less than 90°, wetting occurs. Complete wetting is indicated by a 0° contact angle, and the liquid spreads spontaneously over the solid surface as a thin film. The spreading coefficient S_{SL}, therefore, indicates to what extent a liquid will spread over a solid surface. The work of adhesion, W, provides a measure of the energy necessary to separate the liquid and the solid at the interface. Additional factors must be considered when the bond strength between solid metal and oxide is studied.

Since the interfacial energies of the various phases play a dominant role in the wetting and bonding of metal-oxide systems, their effect on bond strength will be included in this investigation. The experimental approach will be described in the next section.

The methods for bonding oxides to metals are legion, and only some of the more common techniques will be described. Several excellent literature reviews (Ref. 5) have been made of ceramic-to-metal seals and the procedures used are essentially the same; viz., that ceramic surfaces are usually first metallized in order to promote wetting and bonding with the desired metal matrix or seal. This is usually accomplished by one or more of several techniques (Ref. 5b, 5c, 6):

A. SOLID, LIQUID AND VAPOR UNDER EQUILIBRIUM CONDITIONS



(1) WETTING, $\Theta < 90^{\circ}$ (2) NON-WETTING, $\Theta > 90^{\circ}$

B. SYMBOLS

γ = surface energy (ergs/cm^2)
for liquids; surface tension (dynes/cm)

Subscripts

S, L, V = solid, liquid, vapor

Θ = contact angle between solid-liquid and liquid-vapor interfaces

S = spreading coefficient; if S is positive, liquid will spread over solid surface

W = work of adhesion; the energy required to separate the liquid and solid at the interface

C. RELATIONSHIPS

$$\gamma_{SL} = \gamma_{SV} - \gamma_{LV} \cos \Theta \quad (1)$$

$$S_{SL} = \gamma_{SV} - \gamma_{LV} + \gamma_{SL} \quad (2)$$

for spreading; $\gamma_{SV} > \gamma_{LV} + \gamma_{SL}$ (3)

$$W = \gamma_{SV} + \gamma_{LV} - \gamma_{SL} \quad (4)$$

or $W = \gamma_{LV} (1 + \cos \Theta)$ (5)

Figure 2. Surface Energy Relationships Between Solid, Liquid and Vapor Interfaces

(a) Metallizing Paints - Paints of Au, Ag, Pt and Cu are frequently applied to ceramic surfaces and serve as a base for soldering or electroplating. Other materials such as Co, Ni, and Mo have also been applied as paints.

(b) Powders and Pastes - Mo, W, Cu, Ni and Fe Powders, with or without other additives (fluxes, glasses, organic vehicles), are commonly used as metallizing media. Mo-Mn powders and various metal powders mixed with Ti or Zr (active metal process) are extensively used to form strong, adherent bonds between many metal-ceramic materials. Glass and metal powders as well as glasses containing Pb, Be, Sn, Zn have also been used as metallizers.

(c) Flame-Sprayed Coatings - Mixtures of WC, Fe, Ni, Co, and Co and Cu powders have been flame sprayed on ceramic surfaces. Generally the coatings are not sufficiently adherent and are too sensitive to process variations.

(d) Vapor Deposition - During the past few years this method has been receiving increased attention. Coating have been applied by decomposition or reduction of chlorides, carbonyls, and other volatile components containing metals such as Ni, Cr, Ti, Si, Mo, W, Ta, Fe, Co and Cu. Frequently further heat treatment or reduction in a hydrogen atmosphere may be required. Vacuum deposition by sputtering and evaporation are also commonly employed to give very thin, continuous metallic films on oxide surfaces.

The bonding of metals to alumina (Al_2O_3) (Ref. 5 & 6) and to sapphire (pure, single crystal Al_2O_3) (Ref. 7) has been investigated extensively. Metallizers which promote the best wetting and bonding are usually the active metals (hydrides of Ti, Zr, Cb and Ta) and Mo or 20% Mn-Mo alloys. As the purity of the Al_2O_3 is increased, the strength of the bond usually decreases. This is exemplified in a study conducted by Cole and co-workers (Ref. 8a), where bond stresses of 28,4000 psi for a 94% Al_2O_3 composition, 22,000 psi for a 96% Al_2O_3 composition, and 22,000 psi for a 96% Al_2O_3 body were measured by a tensile test. All bonds were based on a Mo alloy.

The problem of accurately measuring tensile or shear strengths of metal-to-ceramic bonds is difficult and requires extreme care in preparation and final polishing of the test specimens. In comparison to the literature on hermetic seals, there are far fewer reports (Ref. 8 and 9) dealing with the testing and measurement of specific types of bond strengths. As a result, it becomes necessary to conduct experimental studies in order to develop high strength bonds in specific metal-oxide (whisker) systems. Such a program is described in the next section.

III. EXPERIMENTAL PROGRAM

It is planned to use one metal (nickel) and one oxide (Al_2O_3) system so that a study of the factors affecting bond strength can be studied in greater detail. Rather than using whiskers, because of their small size, it is planned to conduct studies on large single crystals of α- Al_2O_3. The final test results, however, will be obtained on metallic composites reinforced with α- Al_2O_3 whiskers and will be used to evaluate the bonding techniques developed during the course of the investigation.

A. FACTORS TO BE INVESTIGATED

The following factors are considered to have the greatest influence on the bond strength and will be investigated:

1. Metal purity (effect of additives)
2. Metallizing (coating) the oxide surface prior to contact with the matrix metal (Ni).
3. Atmosphere composition and heat treatment during bonding.

1. Metal-Oxide System

Nickel was chosen because of its chemical compatibility with α- Al_2O_3 at elevated temperatures, and because it can serve as a base material for many alloys that can be reinforced with α - Al_2O_3 whiskers for elevated temperature applications.

Single crystal alumina (99.99% α - Al_2O_3) was selected because of its purity and availability. Single crystal specimens are preferable for two reasons: (a) there are no grain boundaries or visible pores to contend with, and (b) they have the same crystal structure as the α- Al_2O_3 whiskers to be investigated.

2. Metal Purity (Effect of Additives)

It is known that various additives, such as Fe, Co, Cr, Cu, Ti and Zr, to a metal can significantly alter the wetting of an oxide by another metal. For example, Kurkjian and Kingery (Ref. 10) has shown that Ti has a pronounced effect on the wetting (contact angle) between molten nickel and α - Al_2O_3. Titanium lowers the Ni-α- Al_2O_3

interfacial surface energy and some of it is presumed to be in the oxidized (TiO) state. However, the correlation between wetting and bond strength was not made. For the present study it is planned to add Cr and Ti to pure nickel in various proportions, and to measure their effect on the wetting and bonding of Ni to single crystal α - Al_2O_3.

3. Metallizing (Coating) The Oxide Surface

One method which has been used effectively to promote wetting is that of depositing a metallic coating on the oxide. The effect of wetting and bonding of nickel to sapphire (α- Al_2O_3) by first coating the oxide surfaces with metallic films such as Cr and Ti will be evaluated.

It is planned to metallize sapphire surfaces by sputtering techniques since carefully controlled, high purity, continuous metallic films have been successfully deposited on sapphire surfaces in this laboratory. Subsequent heat treatment can be employed to promote further bonding. The nature of the bonding, uniformity of coating thickness and composition are all subject to greater control than by the other methods cited previously (Section II).

4. Atmosphere and Heat Treatment

The composition of the atmosphere (argon, air, vacuo, etc.) can have a pronounced effect on the wetting and bonding of metal-oxide systems. In the case of gold on SiO_2 surfaces, for example, the presence of oxygen during the period that the metal is molten has a marked effect on developing a strong bond when the metal is solidified (Ref. 9). Iredale (Ref. 11) reported that the effect of atmosphere is more prevalent with materials with appreciable gas solubility. Such is the case with oxygen and silver. Since oxygen also affects the surface tension of liquid nickel, its effect on the wetting of molten nickel on α - Al_2O_3 will be investigated.

B. EXPERIMENTAL PROCEDURE

Two types of experiments will be conducted: (1) those concerned with wetting and interfacial energies (sessile drop tests) and (2) those concerned with bond strength (shear strength tests). These will now be described.

1. Sessile Drop Experiments

Much of the effort will be based on the results of sessile drop experiments, as shown in Figure 3A, since they provide direct information on contact angle (wetting) and surface tension of the liquid metal on $\alpha - Al_2O_3$. Furthermore, these studies will also provide information on the stability (chemical interaction) between the phases at elevated temperatures, and will provide a guide for evaluating the effects of metal additions, of metallizing coatings, and of contaminating gases such as oxygen. By combining this information with analyses of residual stresses due to thermal expansion mismatch between the solidified metal and oxide, and making comparisons with the bond strengths observed, a procedure can be formulated for tailor-making the most suitable bonding systems for whisker-metal composites.

2. Evaluation of Bond Strengths

In order to determine the effects of the various parameters on the bond strength of the Ni - α - Al_2O_3 system, shear (pull-out) and transverse strength tests are planned. One test is based on a 'shear' type test which simulates the conditions in a metal-whisker composite loaded in tension (fiber axis parallel to the tensile load) and the other is based on a transverse rupture test where a rod specimen is used. The specimen is actually bonded together at the center by a metal joint. These tests are shown schematically in Figure 3B. The transverse rupture test was included because it is more precise, has been standardized, and can also provide a means for comparing values cited in the literature. It is planned, however, that the 'pull-out' test will receive the most emphasis since it more closely simulates the conditions in a whisker-metal matrix. A tensile specimen for this test is shown in Figure 4. The other two tests will be used primarily for screening tests.

In addition to strength measurements, X-ray and metallographic techniques will be used to study the composition and structure of the metal-oxide interface. An electron microscope is also available for this study.

3. Ni - α - Al_2O_3 (Whisker) Composites

In order to evaluate the bonding in metal-whisker composites, composite specimens will be fabricated and tested in tension (see Figure 3C). These tests will provide an ultimate means for determining the effectiveness of the bonding techniques developed during the course of the study.

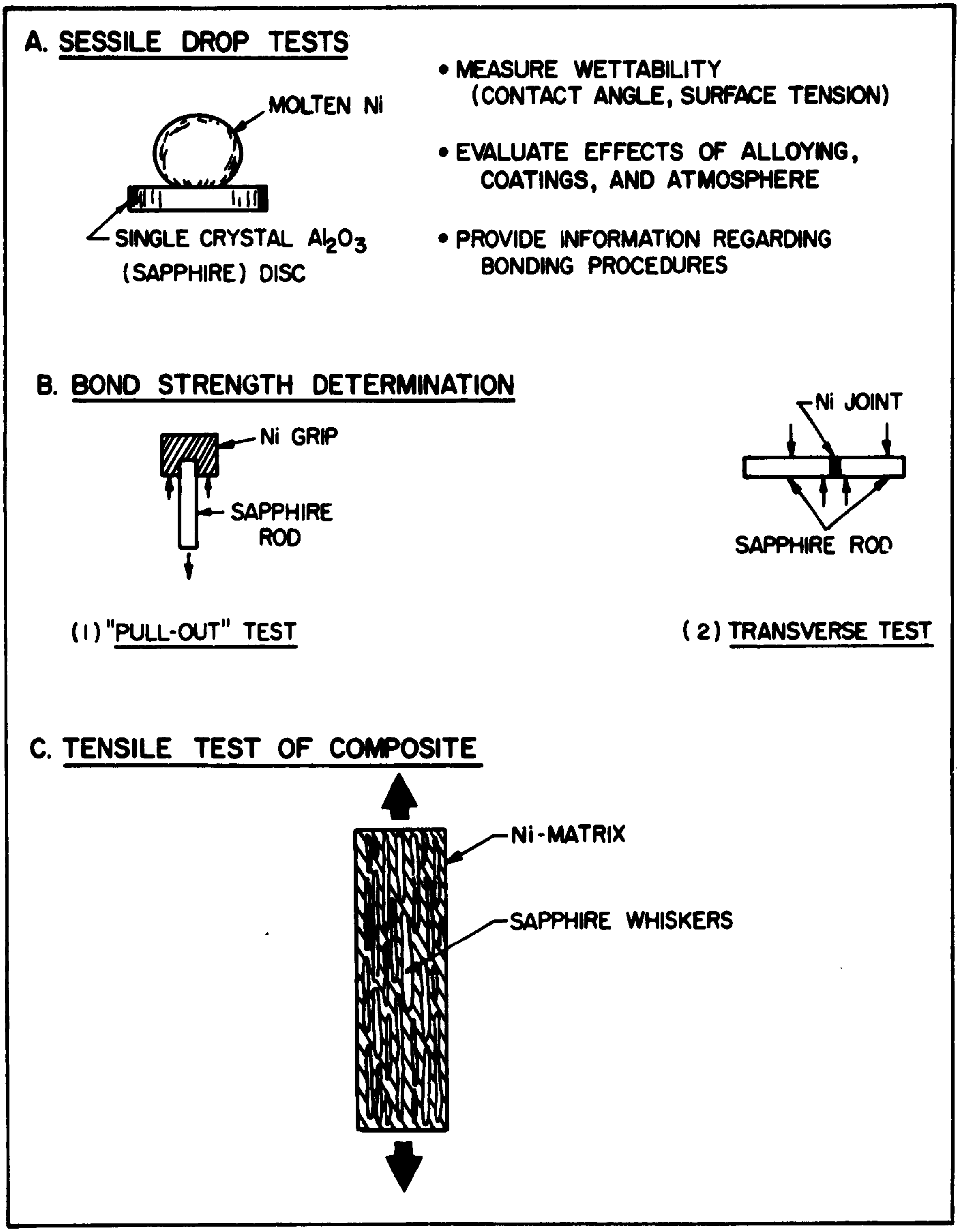


Figure 3. Schematic Diagram of Testing Methods

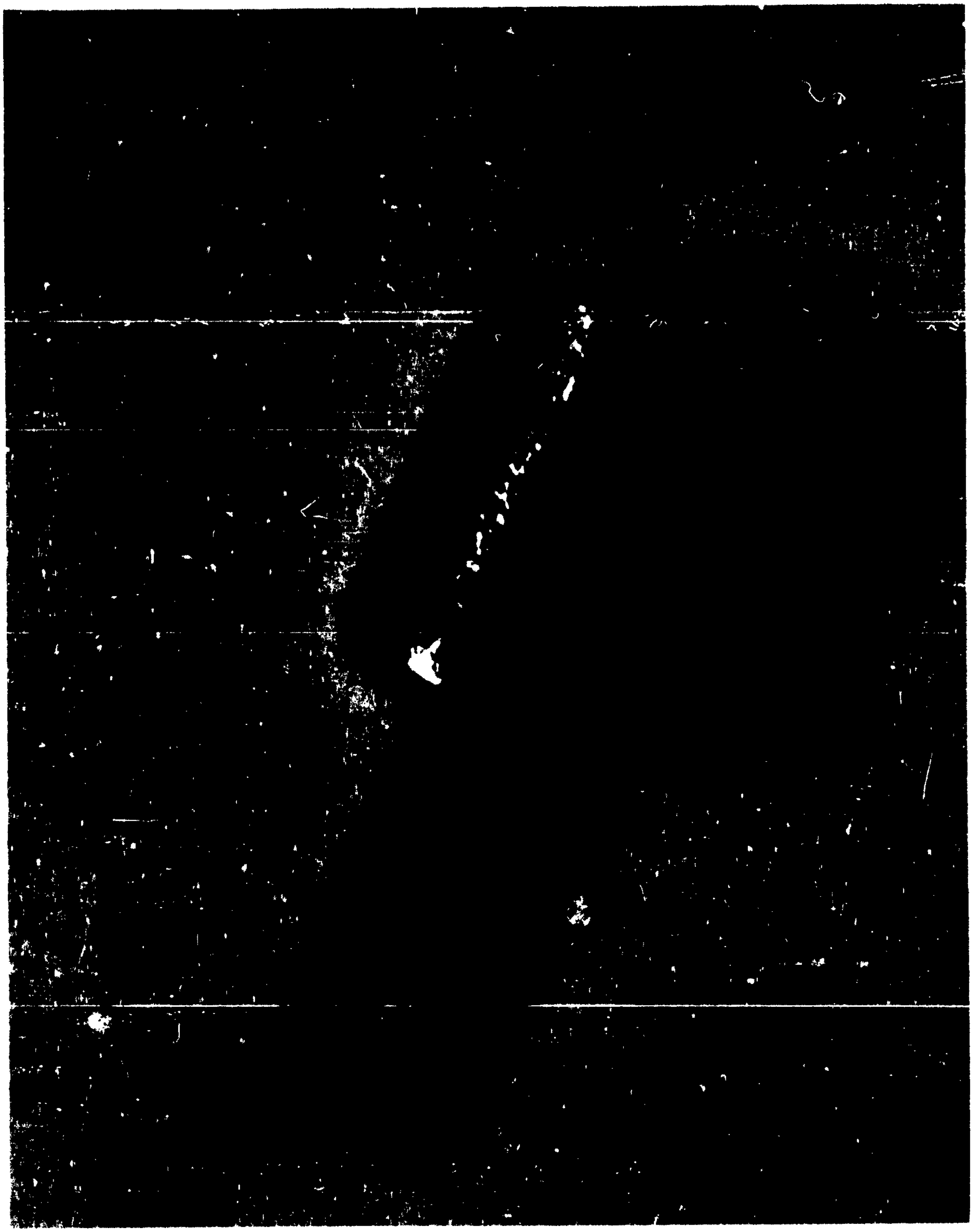


Figure 4. Specimen for 'Pull-out' Test. In this Case, Aluminum Metal Was Cast Onto a Sapphire Rod (3-1/2X)

IV. RESULTS

A sessile-drop apparatus was designed for conducting experimental studies on the interfacial behavior of molten nickel on single crystal α-Al_2O_3. Since the wetting (contact angle) and bonding of the metal to the oxide is extremely sensitive to impurities, great care was utilized in the design so that the impurity effects would be minimized. The plans and drawings were completed, and the various parts are being made in the shop.

The apparatus consists essentially of a horizontal tube furnace, inductively heated, with temperature capabilities up to 2000°C in vacuum or controlled atmospheres. The furnace portion of the apparatus is shown schematically in figure 5. The entire assemblage is spring loaded on a platform with adjustable levelling screws, and this in turn sets on a vibration-free table. A telemicroscope unit with camera attachment was constructed so that a photographic record of the sessile drop can be taken. A commercial two-inch vacuum system with mechanical and diffusion pumps, vacuum gages, and cold trap was ordered.

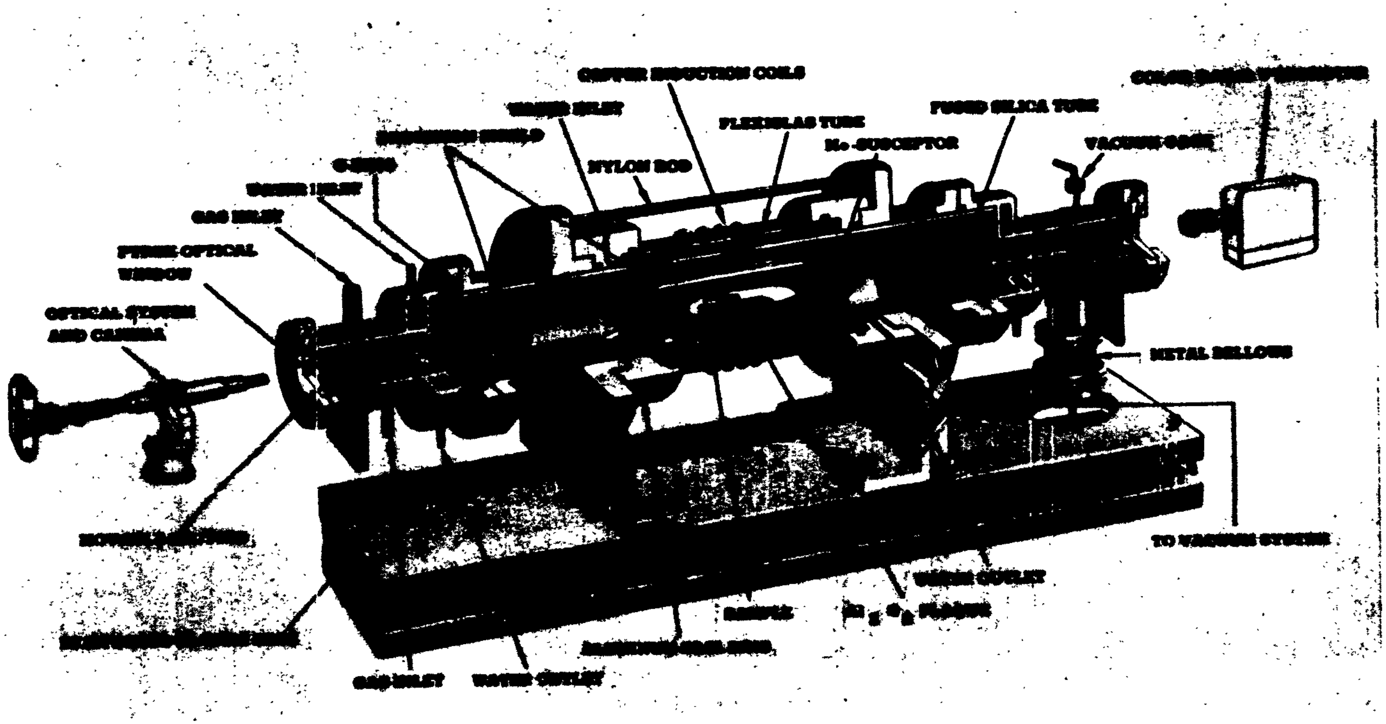


Figure 5. Sessile Drop Apparatus

V. FUTURE WORK

The emphasis on future work will be placed on the early completion of the sessile drop apparatus, and performing calibration runs in order to assess the accuracy of measurement and general performance. The first studies will be based on pure nickel - αAl_2O_3 specimens.

Acknowledgements

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Report No. AMRA CR63-01/1, Nov. 1962, 21 pp-tables-illus, (Contract DA-36-034-ORD-3768RD), DA Proj. 59332008, Unclassified Report.

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