

ELEVATED TEMPERATURE STRAIN GAGES*

J. O. Brittain, D. Geslin and J. F. Lei
Northwestern University

One of the goals of the Host Program is the development of electrical resistance strain gages for static strain measurements at temperatures ≥ 1273 K. Strain gage materials must have a reproducible or predictable response to temperature, time and strain. It is the objective of this research to investigate criteria for the selection of materials for such applications through electrical properties studies. The results of the investigation of two groups of materials, refractory compounds and binary alloy solid solutions are presented in this report.

The materials selection criteria are summarized as follows:

(1) The material should not undergo a phase transformation in the temperature range of interest. This was ascertained by studying existing phase diagrams.

(2) The materials should not undergo any order-disorder transitions or the formation of clusters. In order to satisfy this criteria interstitial refractory compounds were selected for a study of their elevated temperature behavior. In the case of binary alloys an effort was made to locate ideal solid solutions, i.e., alloys in which both elements had activity coefficients close to one (1). In the present case based upon a recommendation of one of our colleagues (2) we set a limit on the activity coefficients of within 15% of one. Thermodynamic data (3,4) suggested that the systems Ag-Pd and Al-V have ranges of concentrations that satisfy this criterion. However, because of the shortage of activity coefficients data, alternative qualitative parameters have been used to select alloys that appear to satisfy this second criterion. Based upon the Hume-Rothery's work, elements that have similar electronegativities and molar volume tend to form ideal solutions. The systems Mo-W, Mo-Re, and Nb-V were selected based upon these qualitative parameters.

(3) Chemical and thermal stability of the materials are important in order to reduce the tendency for oxidation and in order to insure mechanical stability. It was deemed useful to have a high melting temperature in order to minimize recrystallization and grain growth. The importance of insuring elastic behavior over the useful or desired strain range could in principle be achieved by selecting materials for which $(\sigma_{E.L.}/E) > \epsilon_{max}$ where $\sigma_{E.L.}$ is the stress at the elastic limit. E is the Young's Modulus, and ϵ_{max} is the maximum strain to be measured. This parameter will have to be evaluated experimentally due to the lack of published data on the materials under investigation.

(4) Finally, several materials have been selected for the experimental phase of this work based upon published electrical resistivity-temperature data. This was true of the Cr-V alloys for which results were presented last year. Table I listed all of the materials evaluated and the basis for their selection for this program.

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MATERIALS PREPARATION, PROCESSING AND EXPERIMENTAL TECHNIQUE

The alloys listed in Table I have been prepared by arc melting in the usual manner under a purified Ar atmosphere. The 5 to 10 gm homogenized ingots were sliced into thin plates via a diamond cut off disc. Specimens for electrical resistance measurements were processed from the plates by electrical discharge machining. An alternative method of preparing longer and thinner specimens by means of drawing the molten alloy into a quartz tube connected to a vacuum was tried. However, some pick up of Si occurred and we have abandoned this technique.

With two exceptions all of the refractory compounds were prepared as thin films on Al_2O_3 substrate by the various methods listed in Table II. The two exceptions, B_4C and $\alpha\text{-SiC}$, were prepared as bulk specimens by hot pressing and subsequently thinned by slicing into electrical resistance specimens. The refractory compounds thin films were obtained from several sources that have had considerable experience in the various techniques utilized.

The electrical resistance (ER) of the specimens were measured via a four probe technique using Pt or W leads and pressure contacts in conjunction with a ceramic specimen holder. The contacts were checked and found to be ohmic at all temperatures. Measurements were conducted in an evacuated quartz tube under a dynamic vacuum of $\sim 10^{-5}$ torr. Thermal Emf were eliminated by alternating the direction of the current. Initially measurements were conducted on individual specimens; currently three specimens are held in the ceramic device and readings on the three specimens are made by rotating the contacts in the circuit.

RESULTS - REFRACTORY COMPOUND

A. Transition Metal Nitrides

(a) TiN

The ER of two TiN films have been measured, one sample had been prepared by HCD method and the other by CVD method. The electrical resistance-temperature (ER-T) curves are shown in Figs. 1 and 2. We can see that their electrical behaviors are different, the resistance of TiN-1 (HCD) was metallic and increased with increasing temperature while the resistance of TiN-2 (CVD) decreased with increasing temperature like a semiconductor. TiN-2 (CVD) was not as stable as TiN-1 (HCD), however, its TCR at the higher temperatures was much smaller (~ 143 ppm/K) and its reproducibility can probably be improved by thermal cycling. An x-ray analysis indicated that these two TiN films have different lattice parameters and compositions. TiN-1 is TiN with $a = 4.174 \text{ \AA}$ and TiN-2 is $\text{TiN}_{0.9}$ with $a = 4.167 \text{ \AA}$, by using EDAX of SEM, we also found that there are some chlorine (Cl) in the TiN-2 films.

(b) TaN

Two TaN Rf sputtered thin films prepared with different nitrogen (N_2) partial pressures during sputtering (Table I) were tested. The ER-T data of both films displayed metallic behavior, Fig. 3, however, the film with the lower N_2 partial pressure (TaN-1) had a smaller TCR (~ 260 ppm/K) and the TCR of both specimens decreased with thermal cycling. The drift rates were about 0.5%/hr for both films.

(c) ZrN

Three Rf sputtered ZrN specimens were studied. A comparison of these three samples during the second cycle of heating is shown in Fig. 4. The film with the highest ratio of N_2 during sputtering (i.e., ZrN-1) had the lowest TCR (~ 290 ppm/K) but it also had the highest drift rate (DR) ($\sim 0.54\%/hr$).

(d) CrN

The ER-T data for the CrN was metallic with a very large TCR (~ 710 ppm/K). The drift rate of resistance was also very large ($\sim 20\%/hr$). This may have been due to the oxidation of CrN and the formation of a Cr_2O_3 layer that may not have been protective. Figure 5 compares the resistance-temperature data for four different nitrides, clearly TaN has the lowest TCR while CrN has the highest TCR.

B. Transition Metal Carbides

TiC

The results of measurements on Two TiC films are shown in Figs. 6 and 7 respectively. Just like TiN, the film prepared by CVD method (TiC-2) had the higher drift rate at 1275 K ($\sim -0.5\%/hr$) but a lower TRC (~ 250 ppm/K).

C. Semiconductor

(a) B_4C

Among all the specimens studied, the results obtained for the B_4C specimen are most promising: (1) the smallest TCR (~ 160 ppm/K) and (2) the smallest drift rate ($\sim 0.1\%/hr$ for 17 hours) as shown in Fig. 8. Additional experiments are planned for B_4C .

(b) SiC

Figure 9 shows the ER-T result of a β -SiC film prepared by the EBE method on an Al_2O_3 substrate (SiC-1), its TCR at 1273 K is ~ 330 ppm/K and the drift rate is $\sim 0.3\%/hr$. This high drift rate may be reduced by increasing the thickness of the film in order to minimize the apparent evaporation effect. ER-T measurements in an Argon atmosphere are planned in order to minimize any effects that may be associated with the high vacuum utilized in the present measurements. Future plans will also include measurements in an oxidizing environment with the β -SiC protected by a nonconducting high temperature cement. The hot pressed high purity α -SiC was more stable, with a drift rate of only $\sim 0.04\%/hr$ but a larger TCR (~ 1940 ppm/K), Fig. 10. It may be possible to decrease the TCR in the α -SiC by doping.

SUMMARY - REFRACTORY COMPOUNDS

The results for all the specimens are summarized in Table III. Based upon the target values for TCR of 200 ppm/K and a drift rate of $\sim 0.1\%/hr$ (5) only B_4C meets the target values. However, for a total error of 10% (6), even this drift value appears to be too large. However, TaN-1 (Rf sputtered), TiN-2 (CVD), TiC-1 (ARE), TiC-2 (CVD) and β -SiC (EBE) also warrant further investigation. Since the ratio of the non-metal atoms to the metallic atoms play an important role in the electrical properties of the transition metal compounds (7), future work will involve this

parameter in the search for strain gage materials with low TCR and high electrical resistance stability, i.e., low drift rate at high temperatures.

RESULTS - BINARY ALLOYS

A. Silver-Palladium System

Based upon the ideal-solid solution criteria we anticipated reproducible and stable electrical resistance-temperature data for alloys with Ag content of less than 5 w/o. Experimental confirmation was reported last year but the TCR at 1273 K of that composition was large. An increase of the Ag content from 4.1 to 15.5 w/o reduced the TCR from 774 ppm/K to 260 ppm/K without a loss of reproducibility or stability of the electrical resistance, Figs. 11, 12, Table IV. With a further increase in the Ag content to 35.3 w/o Ag, the composition known to produce an even lower TCR (< 100 ppm/K) a large drift of the resistance at 1273 K occurred, Table IV.

B. Aluminum-Vanadium System

As reported last year an Al-V alloy 79.3 w/o V had a very low but negative TCR (-22 ppm/K) and a low drift (50 ppm/K) at 1273 K. This alloy had been prepared by drawing the molten alloy into a quartz tube. A microprobe analysis indicated the presence of Si. Three additional specimens with higher V contents were prepared by conventional arc melting. The results of the electrical measurements are shown in Figs. 13, 14. The (Er-T) curves were metallic in form and the TCR for the three alloys improved as the V content increased. A ternary alloy of Al-82.1 w/o V-2 Si was prepared to verify the effect of Si additions, the resulting Er-T curves, Fig. 15 was of a different form from the binary alloy, the effect of Si was quite pronounced. This alloy had good TCR and drift characteristics. Further work on the ternary system is planned.

C. Niobium-Vanadium System

The composition 5.2 w/o V was selected for investigation based upon the reported good oxidation resistance (8). While the reproducibility after the first heating cycle and the agreement in the Er-T curves for three specimens were good, Fig. 16 and 17; the TCR was large (~ 550 ppm/K) and the drift at 1273 K of 500 ppm/K was larger than desired. It may be that similar to the Al-V system a ternary addition might result in a lower TCR and drift rate.

C. Molybdenum-Rhenium

Based upon published data (9) the 71% Mo alloy was selected to provide an acceptable TCR. The Er-T curve was quite reproducible after the first heating cycle and the drift at 1273 K was quite low (< 80 ppm/hr), however, the TCR of (623 ppm/K) was higher than desired (Fig. 18). Here again there is the potential to improve the TCR by a ternary addition.

SUMMARY - BINARY ALLOYS

With the exception of the Al-V-Si system, the alloys investigated thus far behave as predicted in terms of reproducibility between cycles after stabilization if the drift at 1273 K is neglected. In an effort to meet the TCR requirement of

< 300 ppm/K, several of the alloys show promise. These alloys are the 15.6 w/o Ag and 35.5 w/o Ag-Pd alloys and the Al-V alloys. While the 35.5 w/o Ag-Pd alloy does not meet our selection criterion as an ideal solution, the low TCR indicates that further study may be of interest. It is also possible that improvements in the electrical behavior of the Nb-V and Mo-Re alloys can be achieved by the addition of third elements.

In order to improve the oxidation resistance of several of the alloys an effort is underway to prepare alloys via mechanical alloying with the inclusion of Yttrium or Cerium oxide. The oxide additions may also serve to stabilize the structure, especially grain size, and improve the electrical behavior at elevated temperatures.

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TABLE I
MATERIALS SELECTED FOR EVALUATION

Material	Basis for Selection
Pd-Ag	1
Al-V	1
Nb-V	2
Cr-V	3
Mo-Re	2, 3
TiN	4
TiC	4
ZrN	4
TaN	4
CrN	4
B ₄ C	4
SiC	4

1. Ideal solution, thermodynamic data.
2. Qualitative parameters, tendency to form ideal solutions.
3. Resistance-temperature data.
4. Refractory compounds, high melting temperature, structural stability, oxidation resistance.

TABLE II
PREPARATION METHODS FOR REFRACTORY COMPOUNDS

Specimen	Preparation Method	Remarks
TiN-1	HCD, Hollow Cathode Discharge	
-2	CVD, Chemical Vapor Deposition	
TaN-1	RfS Radio Frequency Sputtering	with N ₂ /Ar + N ₂ ~ 22 %
-2		with N ₂ /Ar + N ₂ ~ 25 %
ZrN-1	RfS, Radio Frequency Sputtering	with N ₂ /Ar + N ₂ ~ 1.65%
-2		~ 0.19%
-3		~ 0.16%
CrN	HCD	
TiC-1	ARC, Activated Reactive Evaporation	
-2	CVD	
B ₄ C	Hot Pressed	Bulk Sample
β-SiC-1	EBE, Electron Beam Evaporation	
α-SiC-2	Hot Pressed	Bulk Sample

TABLE III

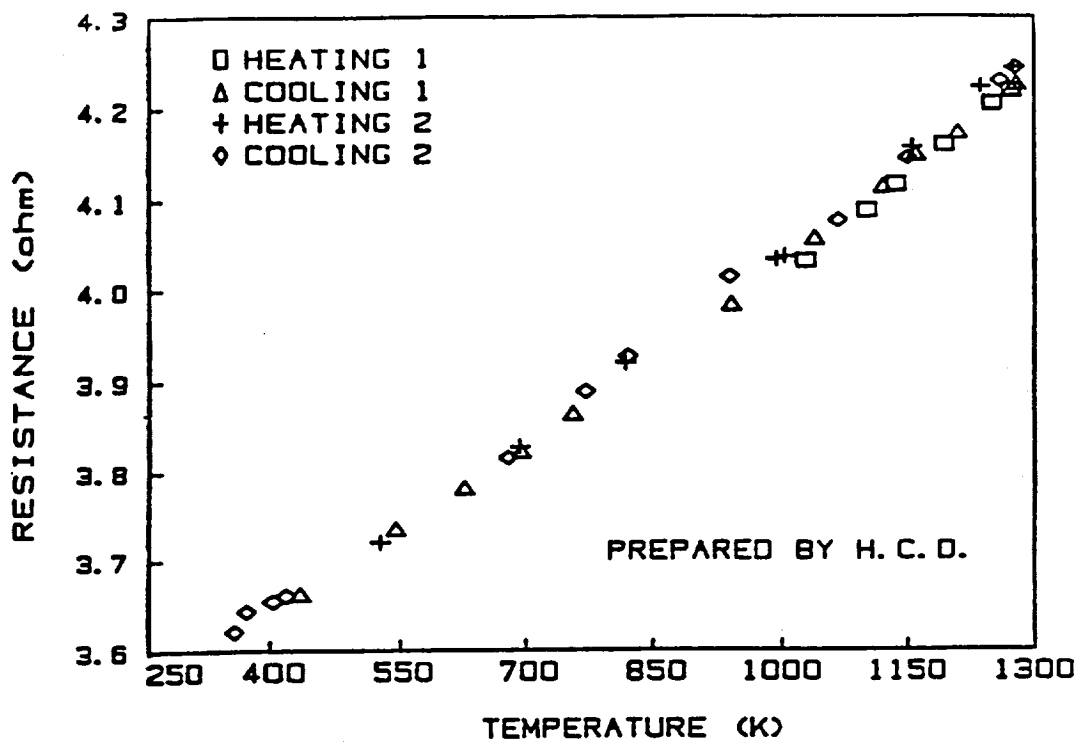
SUMMARIZED RESULTS FOR REFRACTORY COMPOUNDS

<u>Specimen</u>	<u>TCR (ppm/K)</u>	<u>Drift Rate (ppm/hr) (DR)</u>	<u>Remarks</u>
TiN-1 (HCD)	588	1400	
TiN-2 (CVD)	143	2200	Reproducibility improved by thermal cycling
TaN-1	260	4900	TCR and DR decreased with thermal cycling
-2	300	5000	
ZrN-1	290	-5400	TCR increased while DR decreased with cycling.
-2	330	-2600	
-3	420	600	
CrN	710	20%	
TiC-1 (ARE)	366	580	
TiC-2 (CVD)	250	-5000	TCR increased while DR decreased with cycling
B ₄ C	160	940	
β -SiC-1	330	-3000	DR decreased by increasing thickness
α -SiC-2	1940	380	

TABLE IV

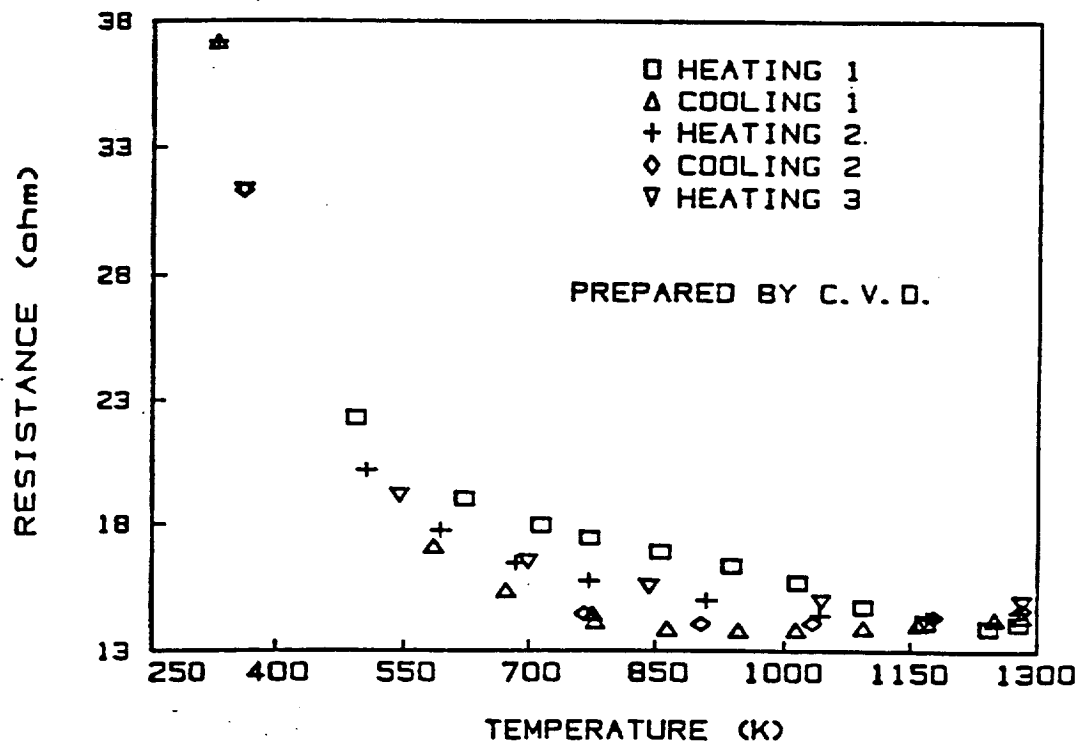
SUMMARIZED RESULTS FOR ALLOYS

<u>Sample w/o</u>	<u>TCR (1270 K) ppm/K</u>	<u>Drift (1270 K) ppm/hr</u>
AgPd 4.3 Ag	774	1723
11.7	460	62
15.5	260	140
35.3	30 to 165	7000 to 38,000
AlV 79.3 V	-22	50
86.65	150	1280
88.9	134	6000
82.1 - 2Si	-20	-1800 cycle 1 - 800 cycle 2
NbV 5.2 V	550	800
CrN 67.4 V	556	1130
MoRe 71 Mo	623	60 cycle 1 75 cycle 2



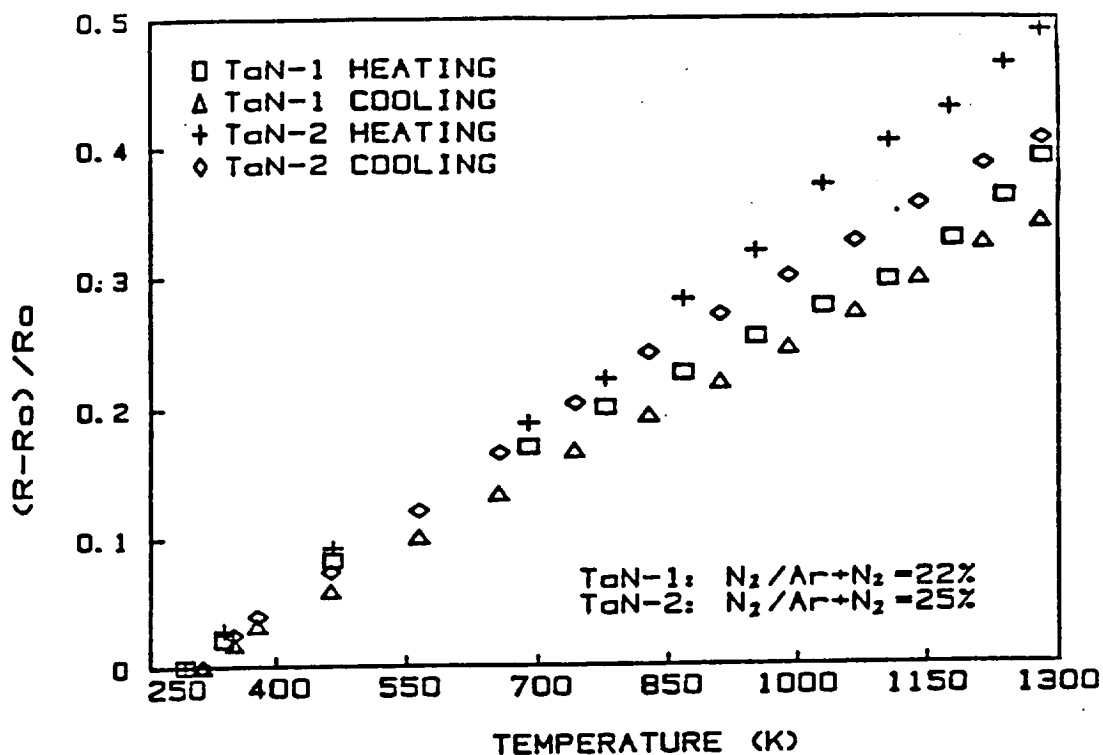
RESISTANCE Vs. TEMPERATURE OF T1N-1

Figure 1



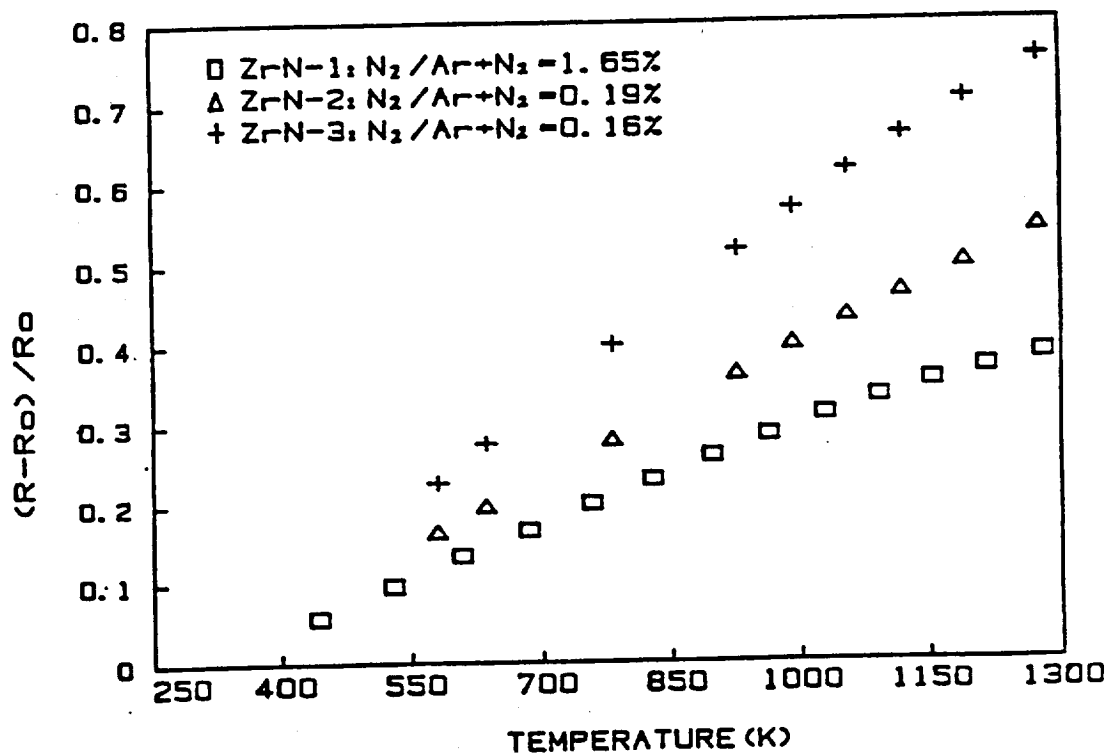
RESISTANCE Vs. TEMPERATURE OF T1N-2

Figure 2



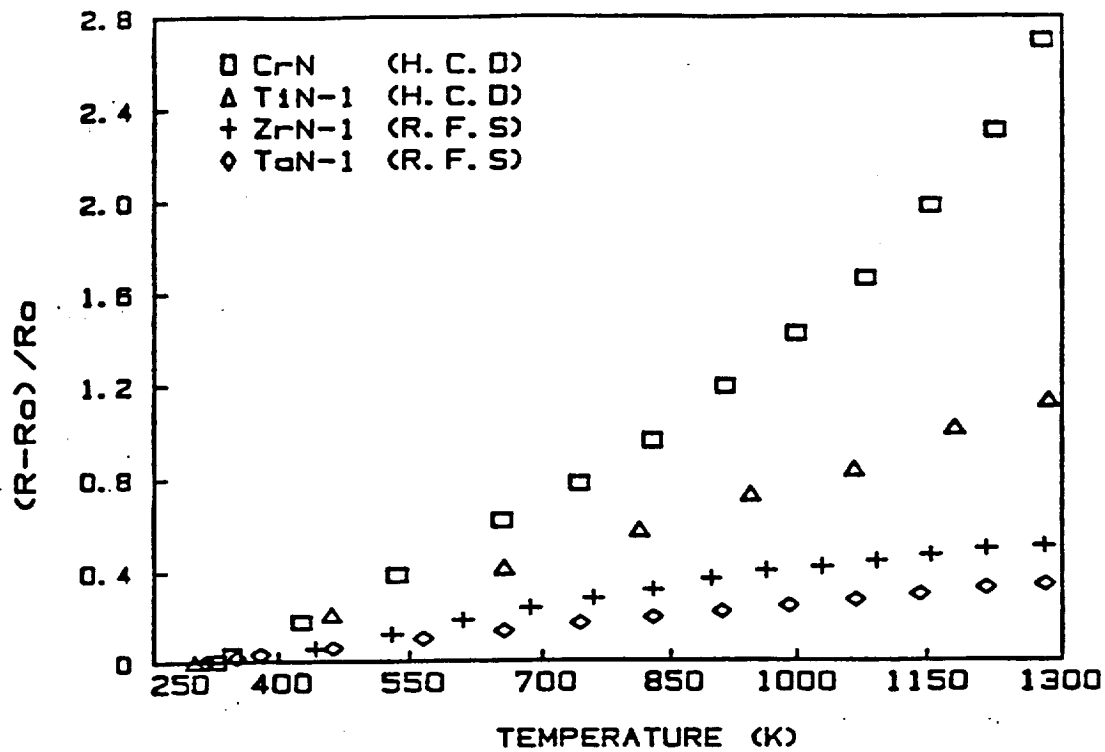
Comparison of Resistance vs. Temperature of two TaN films

Figure 3



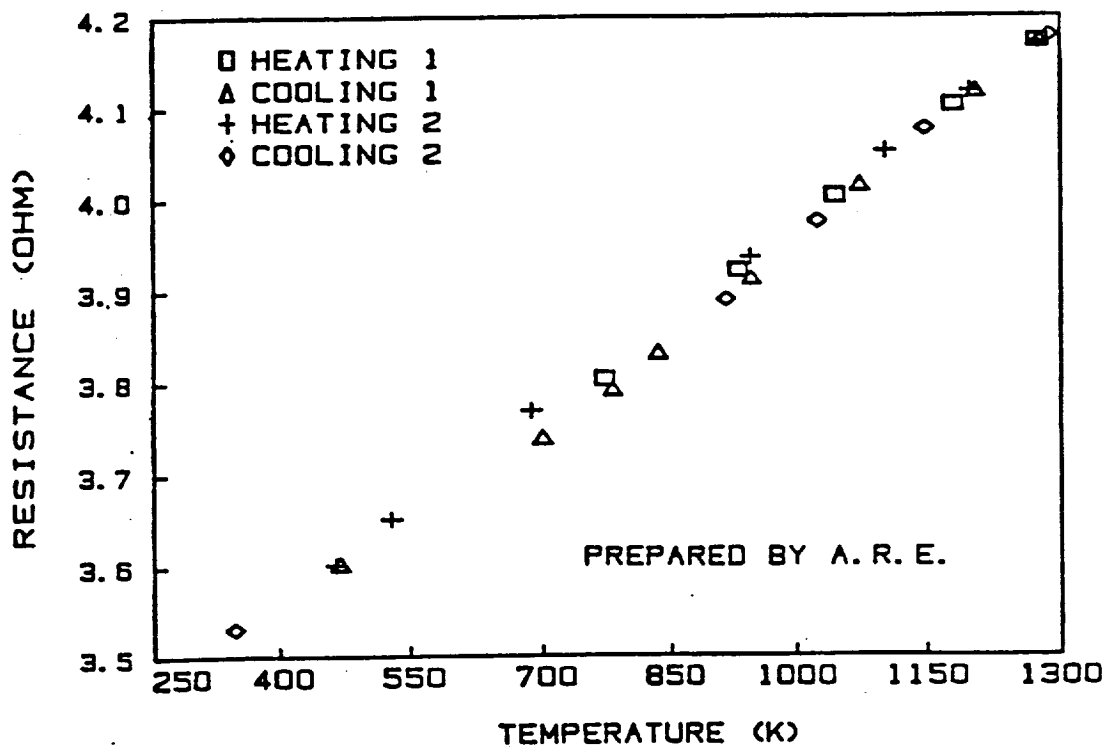
Comparison of Resistance vs. Temperature of three ZrN film

Figure 4



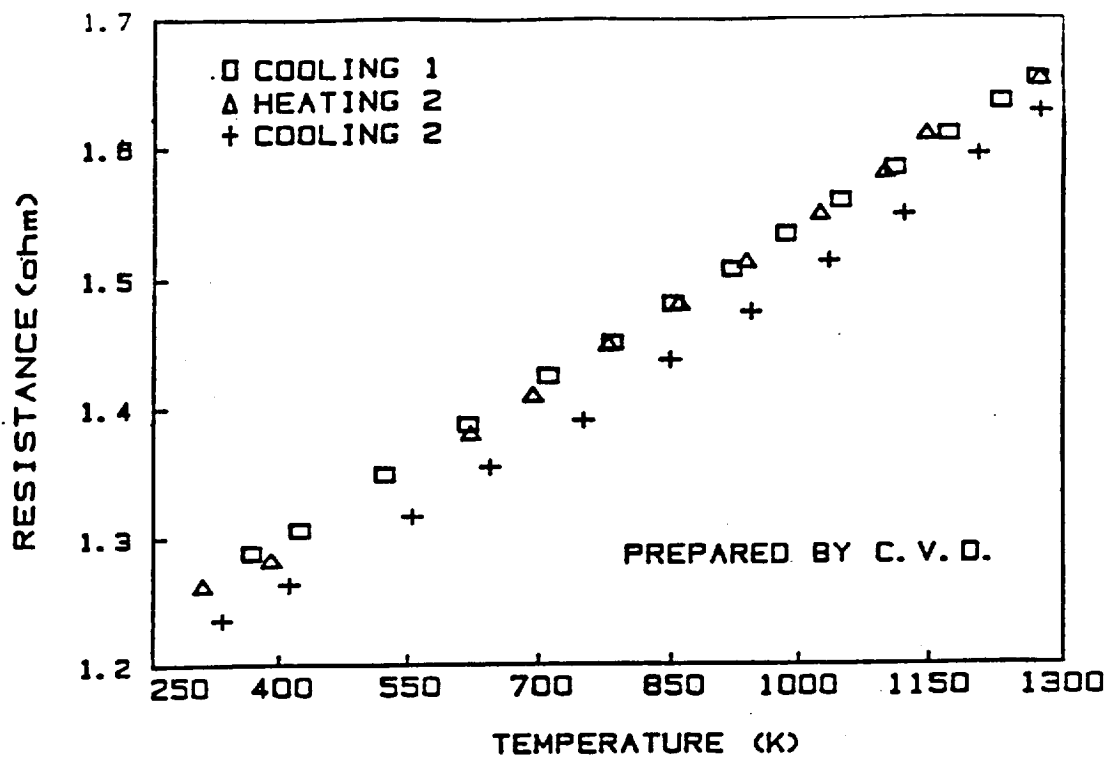
Comparison of Resistance vs. Temperature of four Nitrides

Figure 5



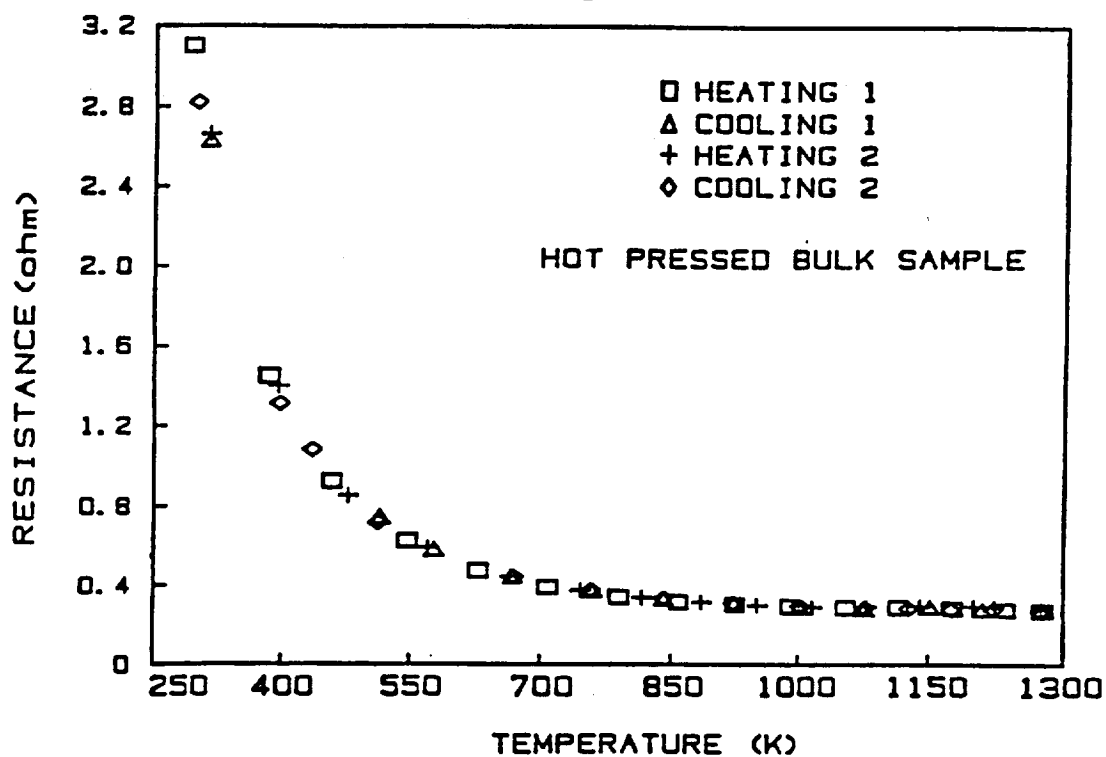
RESISTANCE Vs. TEMPERATURE OF TiC-1

Figure 6



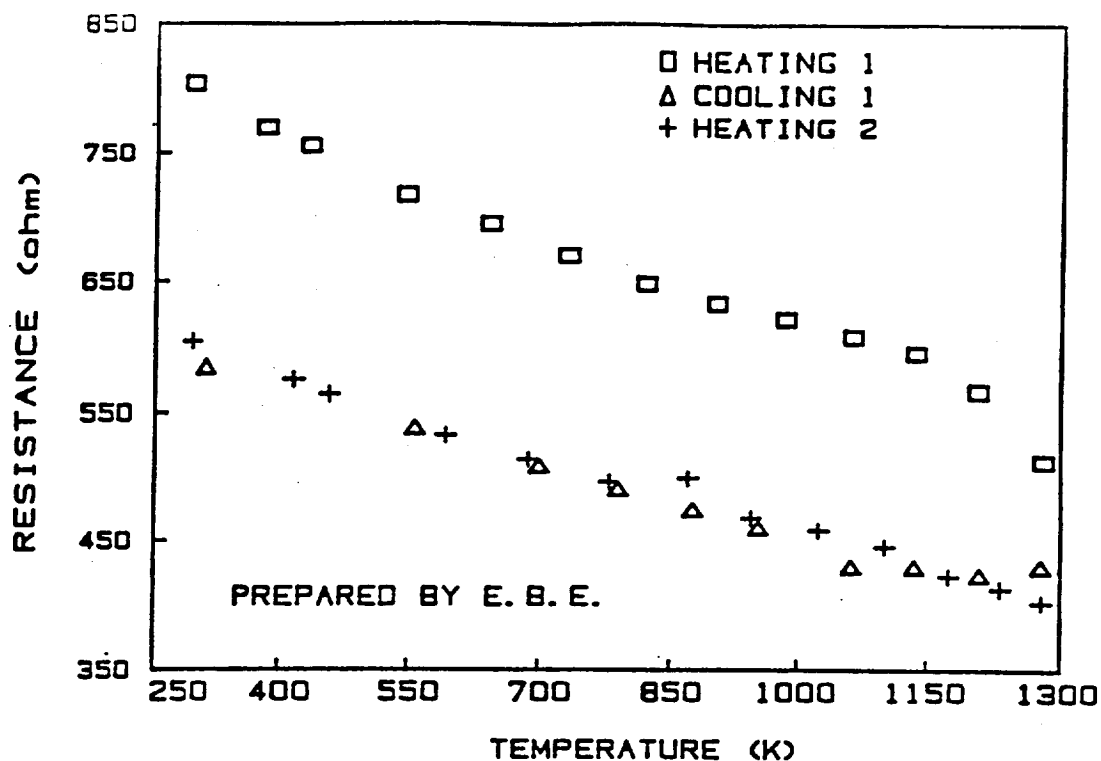
RESISTANCE Vs. TEMPERATURE OF TiC-2

Figure 7



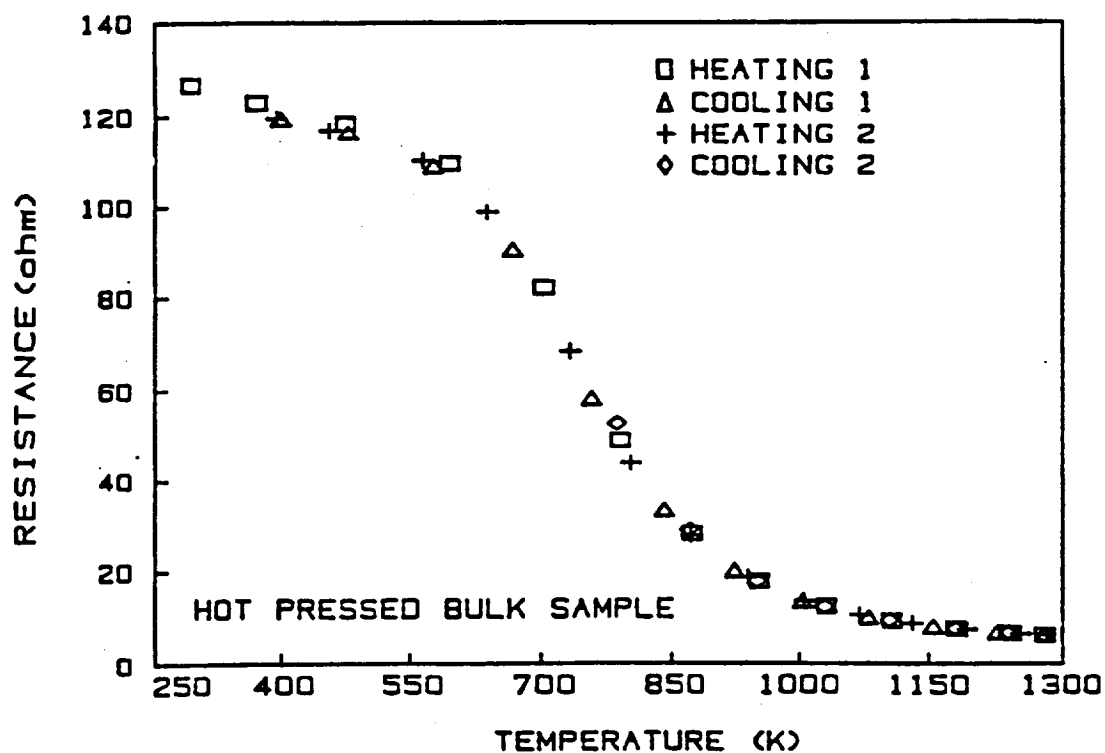
RESISTANCE Vs. TEMPERATURE OF B₄C

Figure 8



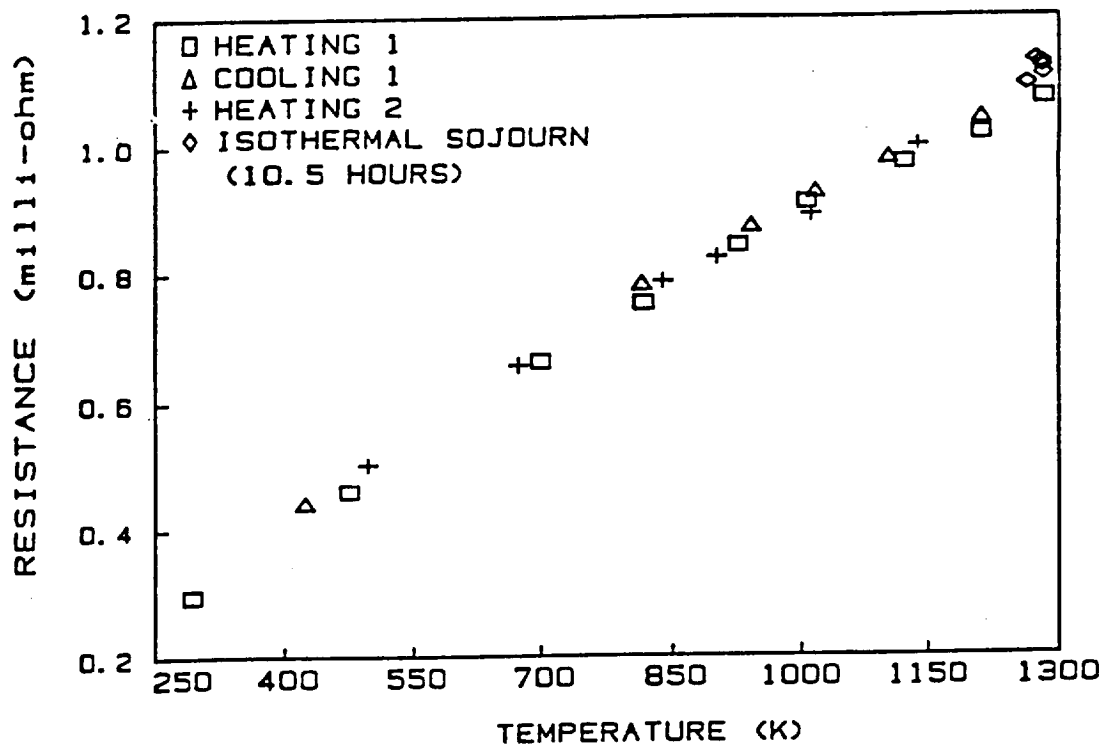
RESISTANCE Vs. TEMPERATURE OF β -SiC-1

Figure 9



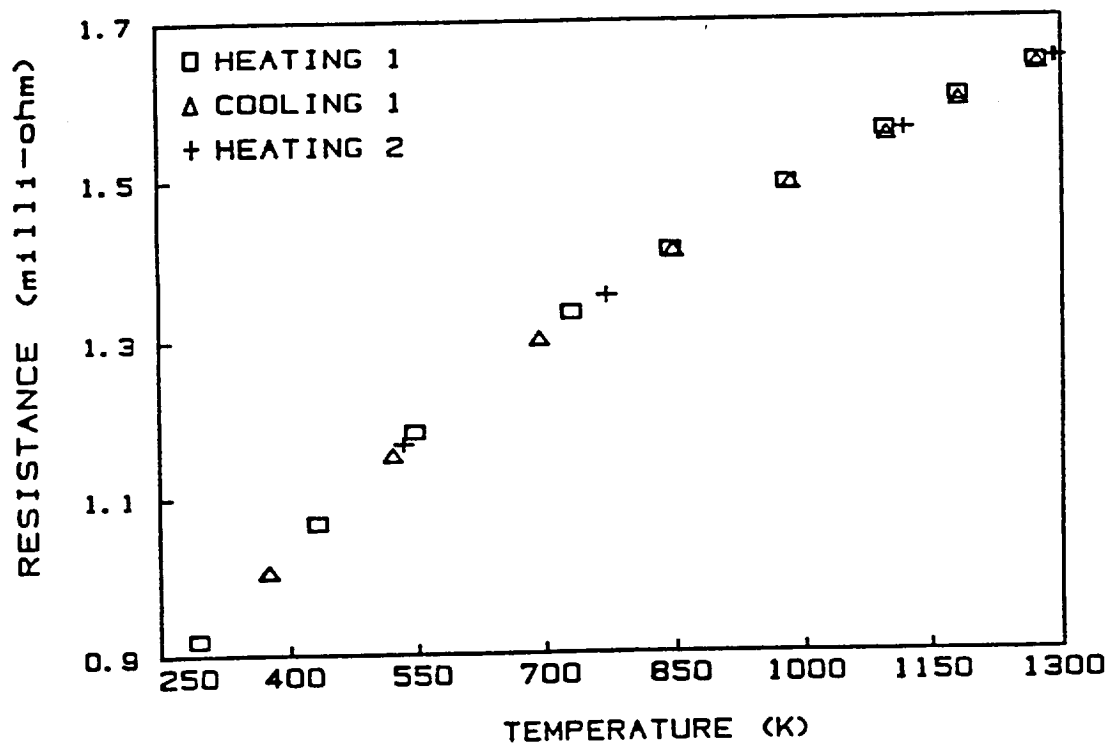
RESISTANCE Vs. TEMPERATURE OF α -SiC-2

Figure 10



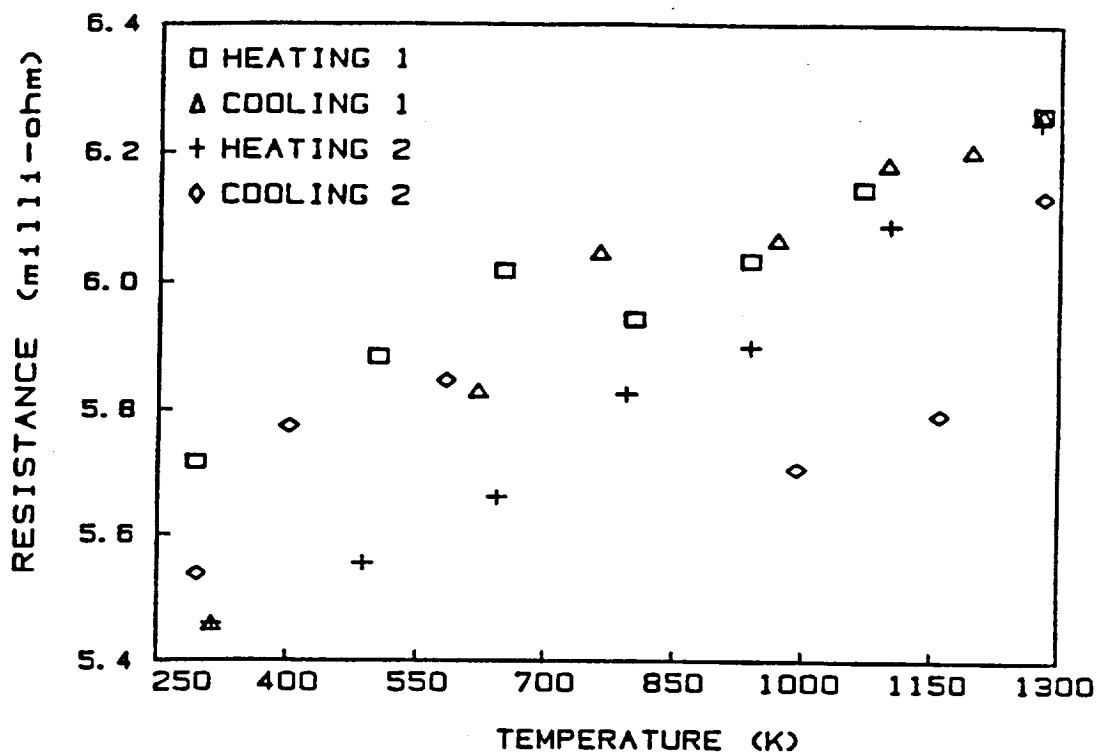
RESISTANCE vs TEMPERATURE of 4.1 w% Ag-Pd

Figure 11



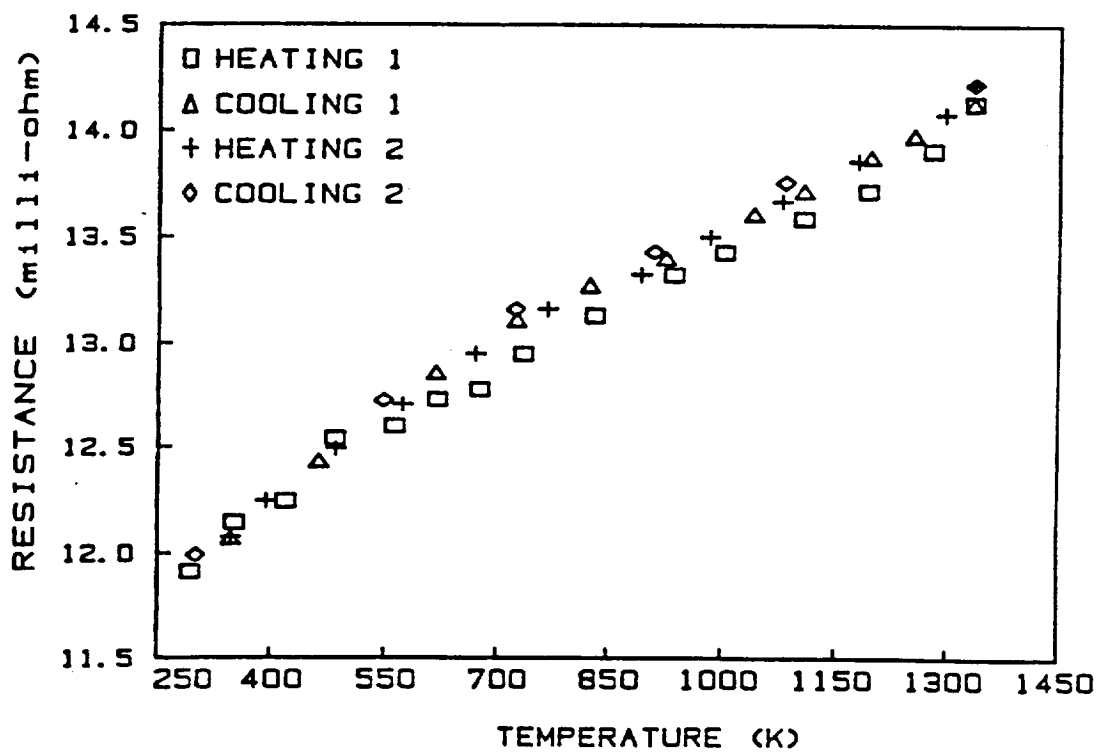
RESISTANCE vs TEMPERATURE of 15.6 w% Ag-Pd

Figure 12



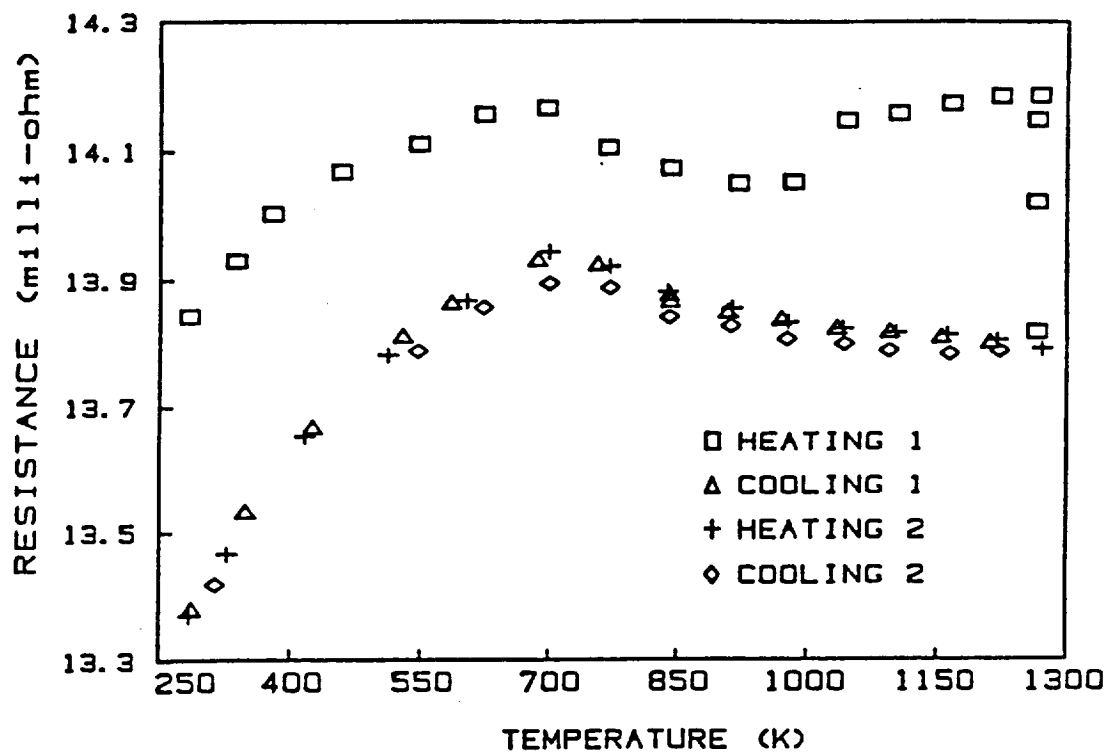
RESISTANCE vs TEMPERATURE of 86.65 wt% V-Al

Figure 13



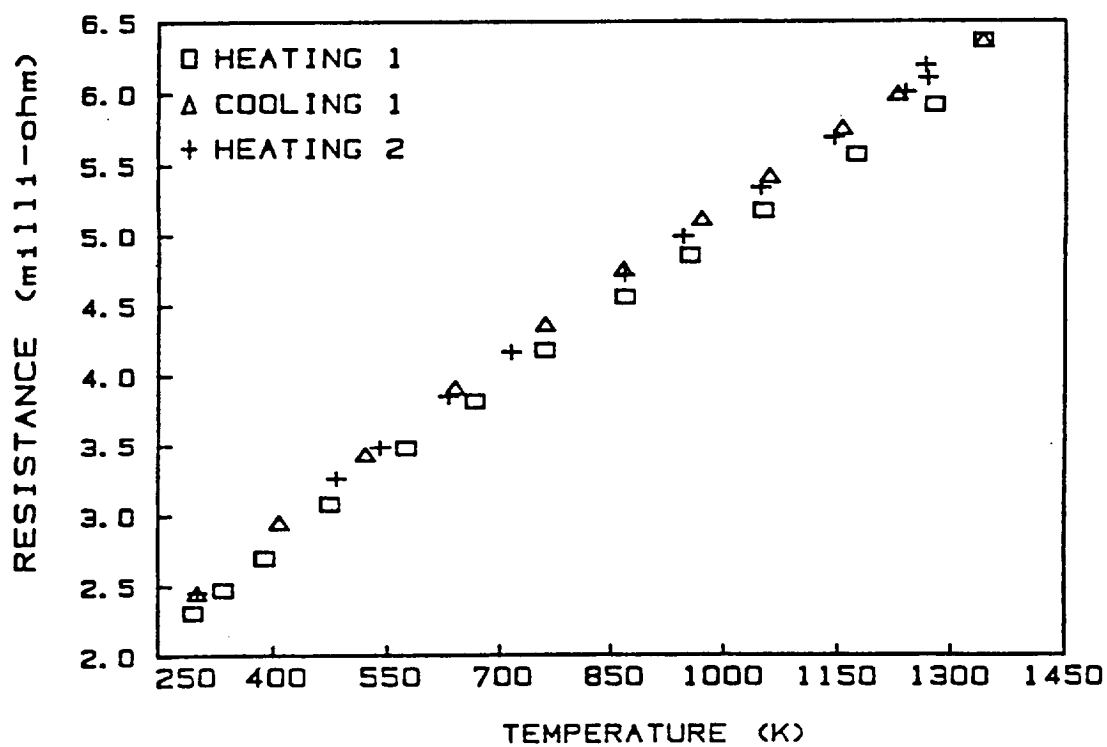
RESISTANCE vs TEMPERATURE of 88.9 wt% V-Al

Figure 14



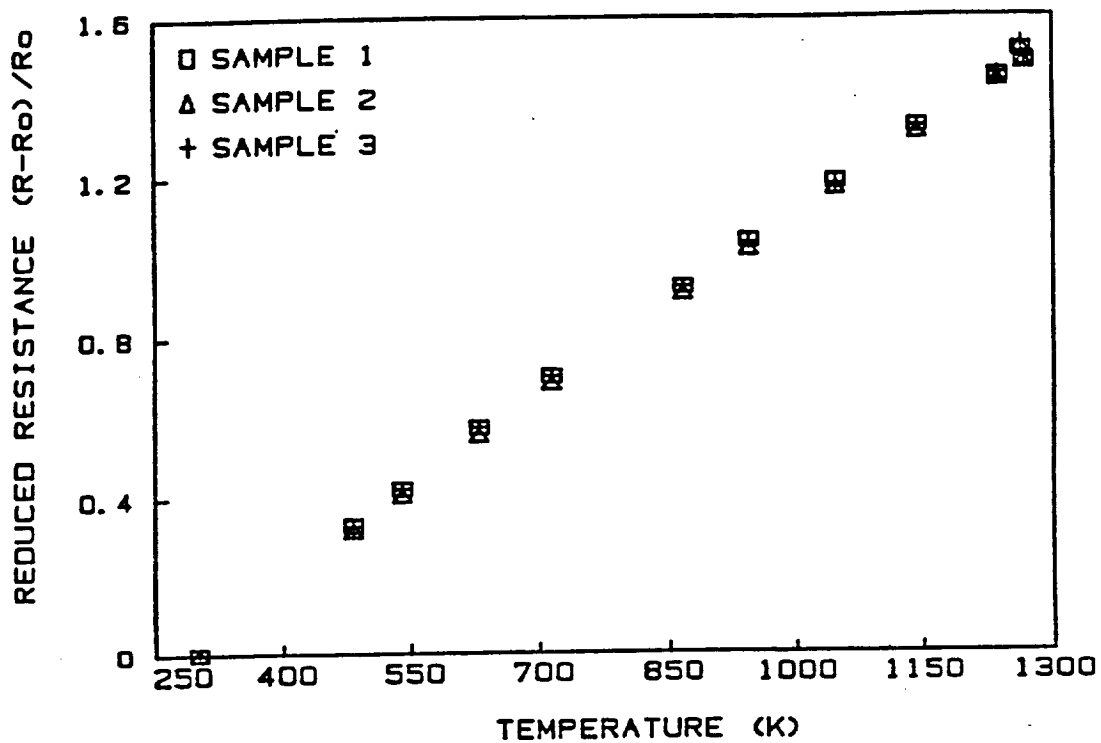
RESISTANCE vs TEMPERATURE of Al- 82.1V- 2Si

Figure 15



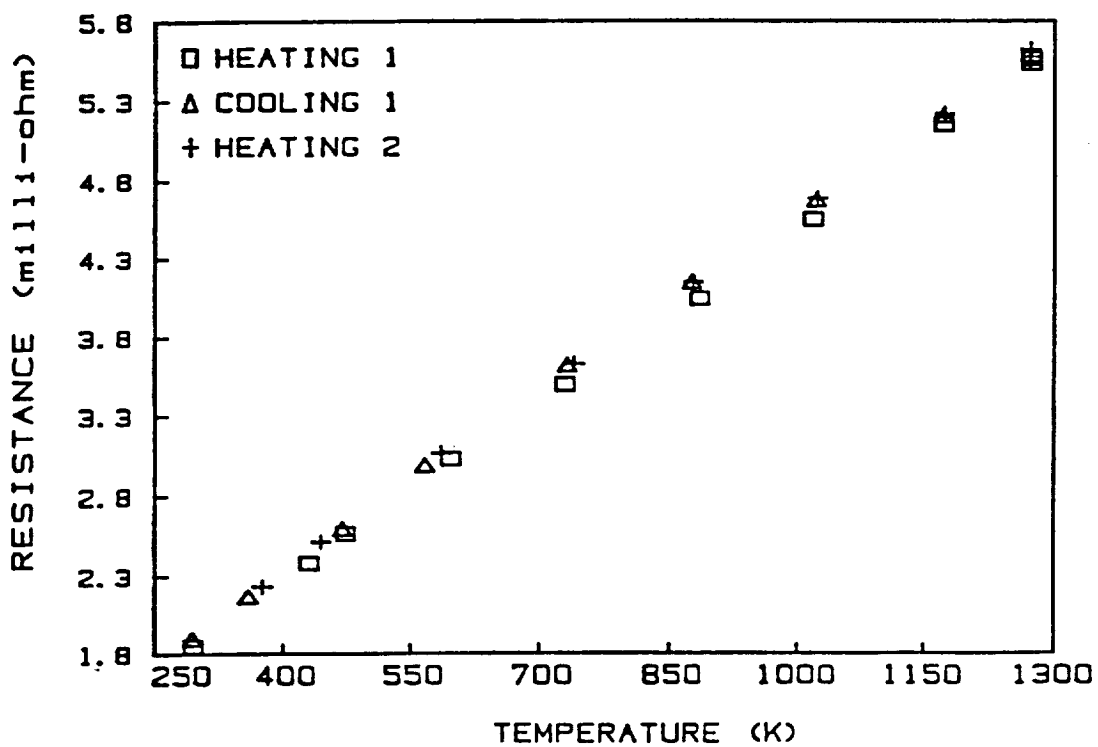
RESISTANCE vs TEMPERATURE of 5.2 w% V-Nb

Figure 16



REDUCED RESISTANCE vs TEMPERATURE for 3 5.2 wt% V-Nb SAMPLES

Figure 17



RESISTANCE vs TEMPERATURE of 71 wt% Mo-Re

Figure 18