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Final Report

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Electrical Properties of Materials for High Temperature Strain Gage Applications

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The objective of this research was to study the electrical resistance of material that are potentially useful as resistance strain gages at temperatures up to 1000 C under static strain conditions. A set of criteria[1] were used to select strain gage candidate materials that are electrically stable and reproducible at all temperatures up to 1000 C. For the experimental phase of this research, the electrical resistance as a function of temperature were studied with three groups of materials: (1) solid solution alloys, (2) transition metal carbides and nitrides and (3) semiconductors. These studies were made in order to identify materials with low temperature coefficients of resistance (TCR) and low resistance drift rates (DR) at 1000 C. A few experiments were made to evaluate the gage factor of several of the more promising candidate materials, B_4C, TiN and TiC. All measurements of electrical resistance were made under vacuum (10^{-5} Torr) via a dc or automatic ac four probes method. Pressure contacts were used and the contacts were checked for ohmicity. Measurements were made at 80°C temperature intervals after the temperature had been stabilized within 1°C. A computerized electrical resistance measurement system was used in the latter stage of the research for automatic recording of the specimen's resistance between 23°C and 1000°C. The furnace temperature was programmed to increase or decrease in 80°C steps

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1 Brittain, J. O. and Lei, J. F., "Elevated Temperature Strain Gages" NASA Conference Publication, 2405, 1985, NASA Lewis Research Center.

with average heating and cooling rates of 1.85^{o}C/min and 0.93^{o}C/min respectively. The results of this investigation are summarized in this report.

Solid State Alloys

In the initial phase of the research, a number of binary alloys were investigated. We reported that the system Ag-Pd, Al-V and Mo-Re appeared to have TCR's and DR's that approached the desired values. In a later phase, third elements were added to these alloys. The selection of the third elements was based upon the Humer-Rothery criteria for ideal solid solutions. All alloys were prepared by arc melting.

A. The Palladium-Silver System

Based upon the results of a study of Pd-Ag alloys with Ag content of 4.1 to 15.5 wt/o, we selected the Pd-12% Ag alloy as the basis for the Ni additions. As indicated in Table 1 and Figure 1, Ni additions had a positive effect on both the TCR and DR.

B. Aluminum-Vandium System

The binary Al-79.3 W+%V was selected as the base composition to study the effects of additions of Cr or Mo. We found that while Mo additions had a positive effect on the TCR and DR., the addition of Cr did not improve the performance of the binary system. Moreover, we noted that while Mo additions resulted in an increase in the TCR, Cr additions resulted in a decrease in the TCR. This suggested that we should try to dope the Al-V system with simultaneous additions of Cr and Mo. There was appreciable scatter in the resistance-temperature data. Annealing at 1000°C-48 hours prior to carrying out the resistance-temperature measurement appeared to improve the data, Figures 2 and 3. Other data for this quatenary alloys showed more scatter when subjected to a shorter anneal at 1000°C. Note that these alloys can be readily fabricated to thin sheets by a combination of hot and warm rolling. Figure 4 summarized the TCR (1000°C) of the Al-79.3 V - 4.25 Mo plus Cr alloy as a function of the Cr addition.

C. Niobium-Vandium System

Based upon the report of good oxidation resistance, the binary alloy Nb-V had been investigated and found to have higher TCR and DR than desired. Additions of Mo to this alloy resulted in a low DR but the Mo additions increased the TCR, Figure 5. It is unfortunate that we did not have the time to investigate the effect of other additions such as Cr on this alloy.

D. Molybdenum-Rhenium-Vandium System

The selection of the Mo-Re system for inclusion in this study was based upon resistance-temperature data found in the literature. The selction of the Mo-20Re alloy for further study was based upon the TCR determined from the published data. In order to reduce the TCR, additions were made of 1 to 7 w/o V. The data presented in Table 1 and Figure 6 shows that the TCR had not been reduced by the V additions, although the DR improved and usually was appreciably better during a second cycle. Figures 7 and 8 show that the R vs T curves were also improved by cycling.

Transition Metal Compounds

Transition metal carbides and nitrides, boron carbide and silicon carbide were selected for the experimental phase of this research. With the exception of several samples of boron carbide and -silicon carbide, all specimens were prepared as thin films. Boron carbide and -silicon carbide bulk specimens were prepared by hot pressing. Samples of TiB_2, $TiSi_2$ as well as B_4C were also prepared as thin films. Tables 2 and 3 summarize the preparation techniques and results of the electrical measurements of the compounds respectively.

The electrical properties of the fourth group transition metal compounds were found to be strongly compositional dependent. The resistivities of the samples increased and their temperature coefficient of resistance decreased with an increase in the vacancy concentration. The films with a high concentration of defects were more unstable at the high temperatures,. This was observed from the drift in the electrical resistance at 1000°C. The presence of both a low TCR and a high electrical resistance stability seemed to be mutually exclusive in this group of materials. Because of their lower temperature dependent resistance and resistance stability, the fourth group transition metal carbide appeared to be better candidates for resistance strain gage applications than their corresponding nitrides. The TCR of the mononitrides decreased as the quantum number of the transition metal increased. For a thin film transition metal compound with resistivities in the range of 900-1000 microohm-cm, a very low temperature coefficient of resistance may be expected. Such films have the potential to be good elevated temperature resistance static strain gages.

The electrical stability of the compound $TiC_{.5}N_{.5}$ was better than that of TiC; however, its TCR was slightly higher than that of TiC. Titanium boride had a very low TCR but its DR was higher than desired but it may be a good candidate for high temperature dynamic resistance strain gage applications. Titanium silicide does not appear to be good for high temperature applications due to its chemical activity at high temperatures. Hafnium nitride and carbide were also investigated. While the electrical resistance-temperature data for one of the HFC thin films appeared to show some promise for gage applications, the variations of the behavior from film to film suggests that preparation techniques may be crucial for this compound, additional studies should be carried out on HfC.

Figure 9 presents the relation between room temperature resistivity and TCR of all the compound studied. As shown, the TCR of the transition metal carbides and nitrides decrease from positive to negative as the resistivities of the materials increased. Note however that TiB_2 and $TiSi_2$ did not follow the other compounds. The behavior depicted in Figure 9 is similar to that observed by Mooij[2] for strongly disordered metallic alloys. The data in Figure 9 suggests that TCR should be zero at a room temperature resistivity of about 800-1000 microohm-cm.

Samples of both hot-pressed bulk and thin film B_4C were

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2 J. H. Mooij, "Electrical Conduction in Concentrated Disordered Transition Metal Alloys", Phys. Stat. Sol., (A)17, 1973, p. 521-530.

investigated under the usual vacuum condition and in the air. B_4C is a degenerate semiconductor with a electrical resistance temperature relation of the form $R=At\exp(E_A/KT)$ with an activation energy, E_A, of about 0.14eV. The TCR and DR of the bulk B_4C at 1000°C was 200 + 10 ppm/°C and 0.095%/hr respectively. The electrical resistance-temperature data for tests conducted in air also had a low TCR and DR, 250 + 10 ppm°/c and 0.9%/h respectively.

The data for -SiC and -SiC are also summarized in Table 4. The -SiC appears to be a more suitable candidate for strain gage applications than the -form. The stability of the electrical resistance of -SiC was dependent upon the film thickness, the thicker films had lower DRs. Also, we observed that doping -SiC with N_2 improved its electrical behavior, i.e. produced a lower TCR.

Gage Factor Measurement

A preliminary study of the piezoresistance effect was undertaken for several of materials that had been selected as promising candidates for resistance strain gages. The change in resistance vs strain at three temperatures for a hot pressed B_4C specimen with a 0.24 ohm-cm room temperature resistivity is shown in Figure 10. Figure 11 shows the gage factor variation with temperature for the same hot pressed B_4C. A drift in the gage factor at 1000°C was found to be 0.22% hour for a period of 6 hours. A comparison of the temperature variation of the gage factor for B_4C was found to be comparable to that observed in the

Fe-Cr-Al-V-Ti- elevated temperature strain gages, Figure 12. We commented that since no effort had been made to optimize the performance of the B_4C strain gage, the results observed were considered favorable for B_4C.

The results of strain measurement on TiC and TiN are as follows: TiC at 254°C G = 3.63, at 525°C, G = 4.08; TiN at 498°, G = 10.9. Unfortunately, difficulties with the electrical contacts thwarted our efforts to extend the measurements to higher temperatures. Other materials that warrant examination of the gage factor are TiC, ZrC, -SiC and TiB_2. Additional studies of HfN and HfC are also warranted. Finally, the promising results of "alloying" some of these compounds such as TiC with N and TiN with C suggest that improvement in the electrical resistance characteristics might be achieved. Figure 9 clearly indicates that these refractory compounds will have a minimum TCR at a room temperature resisitivity of about 800-900 microohm-cm.

TABLE I. SUMMARIZED RESULTS FOR ALLOYS

Alloy, wt.%		TCR (1000°C, ppm/°C) Cycle 2 Heating	Cooling	Cycle 2 Drift (1000°C),ppm/hr)	
Pd-12Ag-Ni	1 Ni	407	424	5080	
	3	392	415	3640	
	5	390	380	1163	
	7	375	394	1460	
Al-79.3V-Mo	.25 Mo	-113	-129	687	
	.75	- 57	- 49	837	
	2.25	- 27	- 31	54	
	4.25	- 20	- 13	103	
Al-79.3V-Cr	.25 Cr	- 28	- 8	- 74	
	.75	- 44	- 80	-176	
	1.25	- 75	-106	130	
	1.75	-107	- 95	170	
	2.25	- 22	- 34	-140	
	2.75	-114	-125	- 70	
Al-79.3V-4.25 Mo-Cr	.05 Cr	- 12	- 18	-110	Annealed 1000°C 2 hrs
	1.25	- 47	- 26	- 30	2 hrs
	2.75	4	12	55	48 hrs
	4.25	38	36	- 87	48 hrs
Nb-8V-MO	1 Mo	436	450	360	
	3	540	550	400	
	5	523	487	484	
	7	557	528	115	
Mo-20 Re-V	1 V	646	644	-1820	
	1	789	768	-3465	
	3	753	753	- 435	
	3	754	743	--	
	5	750	738	- 320	
	5	740	714	- 655	
	7	727	780	- 288	
	7	683	746	- 813	

TABLE 2
Preparation Methods for the Specimens* (MXx)

Specimen	Melting Temp. (^{o}C)	Preparation Methods	Remarks
TiN-1	2930	HCD, Hollow Cathode Discharge	
-2		CVD, Chemical Vapor Deposition	
TiC-1	3250	ARE, Activated Reactive Evaporation	
-2		CVD	
TiCN-1		CVD	N_C $\downarrow +$ N_{N2} $\downarrow -$
-2			
-3			
TiB_2	2970	HCD	with low B_2H_6
$TiSi_2$	1540	RFS	
ZrN-1	2980		N_2/N_2+Ar=0.19%
-2		RFS, Radio Frequency Sputtering	0.18%
-3			0.16%
-4			1.65%
ZrC	3175	ARE	
HfN	3310	HCD	
H_fC-1	3890	ARE	evaporation
-2			time & temp.
-3			were different
TaN-1	3300		N_2/N_2+Ar = 22%
-2		RFS	25%
-3			40%
TaC	3880	ARE	
B_4C		Hot Pressed	Bulk
β-SiC-1		EBE, Electron Beam Evaporation	
-2		CVD	with N_2 dopant
α-SiC		Hot Pressed	Bulk

*all the specimens are prepared as thin films on the Al_2O_3 substrates except for B_4C, α-SiC and CVD prepared β-SiC on Si substrate.

TABLE 3
Experimental Results for the Specimens (MXx)

Specimen	X/M	Resistivity ($\mu\Omega$-cm)	TCR (ppm/^{o}C)	DR (%/hr)
TiN-1	~ 1.0	30.5	588	0.14
-2	0.9 (V_N)	1130	-143	0.22
TiC-1	~1.0	40	338	-0.06
-2	0.8 (V_C)	196	210	-0.50
TiCN-1	C/N=1.0	204	354	0.03
-2	=3.8	213	323	0.43
-3	=49	255	220	0.31
TiB_2		14.6	36	0.35
$TiSi_2$		300	376	0.56
ZrN-1	~ 1.0 (V_N)	180	275	-0.26
-2	<1.0	211	228	-0.36
-3	<<1.0	255	184	1.60
-4	1.1 (V_{Zr})	393	212	-0.54
ZrC	0.9 (V_C)	560	180	-0.13
H_fN	~ 1.0	704	90	0.27
HfC-1	0.95 (V_C)	3735	-426	0.01
-2	0.74	1500	-323	0.50
-3		1120	-110	0.04
TaN-1	<<<1	113	255	0.53
-2	<<1	103	301	0.50
-3	<1 (V_N)	165	438	-0.16
TaC	~ 1.0	72	550	1.50

Table 4

Summarized results for semiconducting candidate materials

Specimen	ρ (Ω-cm)	TCR (ppm/^{o}C)	DR (%/hr)	Remark
B_4C	0.24	-200	0.095	tested in vacuum
		-250	0.9	tested in air
				hot pressed bulk
β-SiC-1	0.01	-330	-0.32	"pure", 0.25 μm thick, prepared by EBE.
-2	0.2	-223	-0.53	with N_2 dopants, 7μm thick, prepared by CVD.
α-SiC	10.1	-2100	0.04	hot pressed bulk

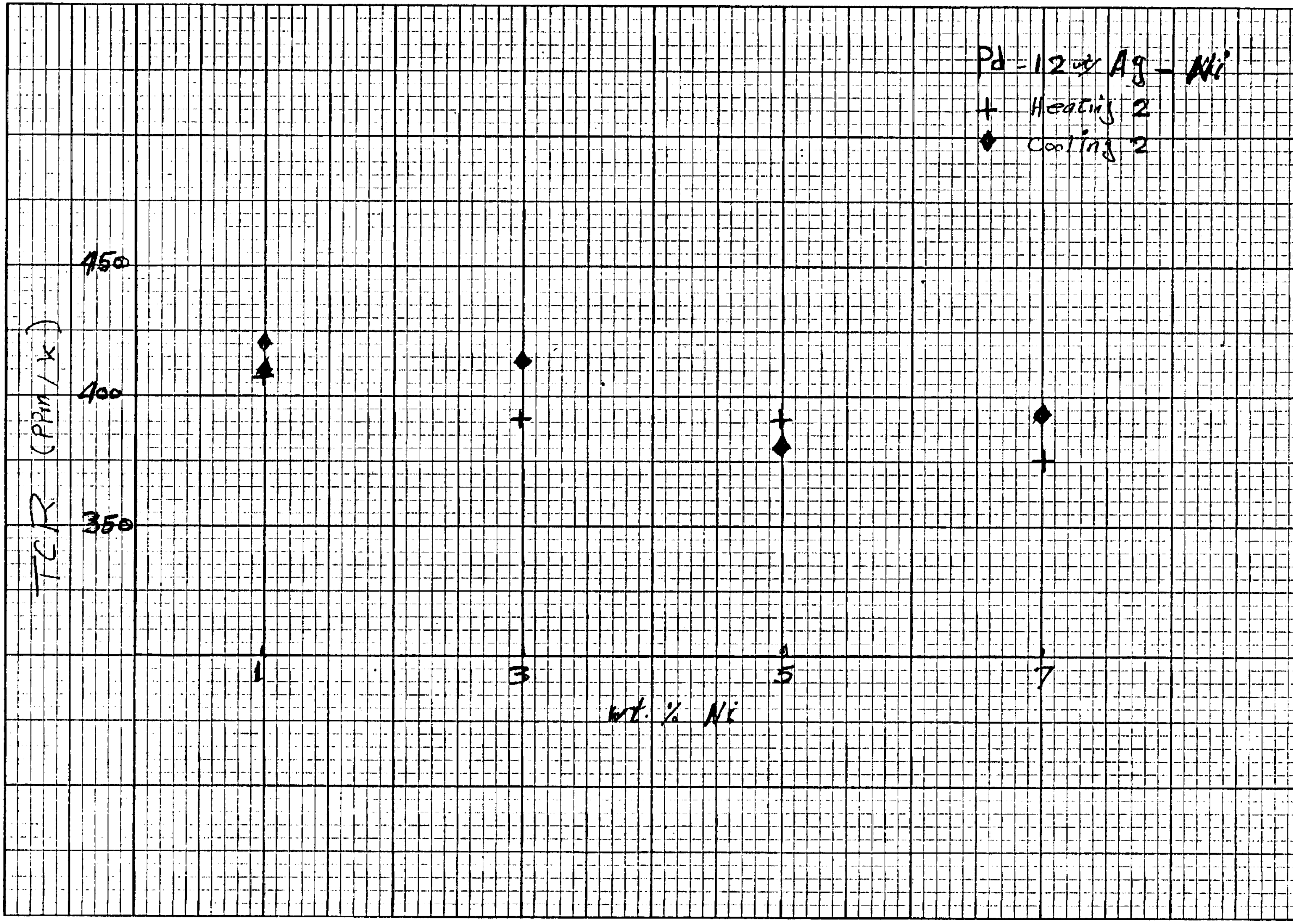

Figure 1

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9270-1006
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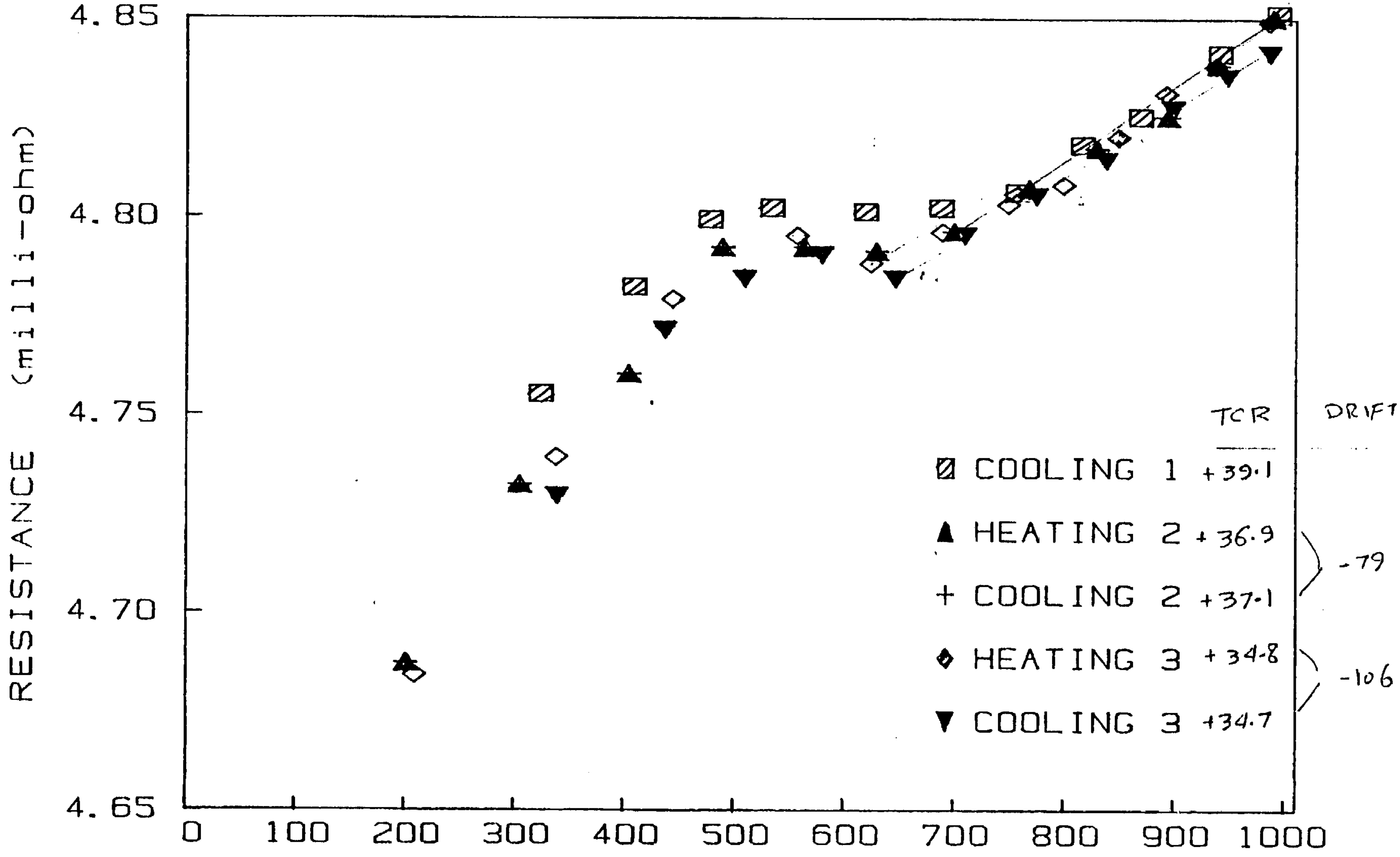
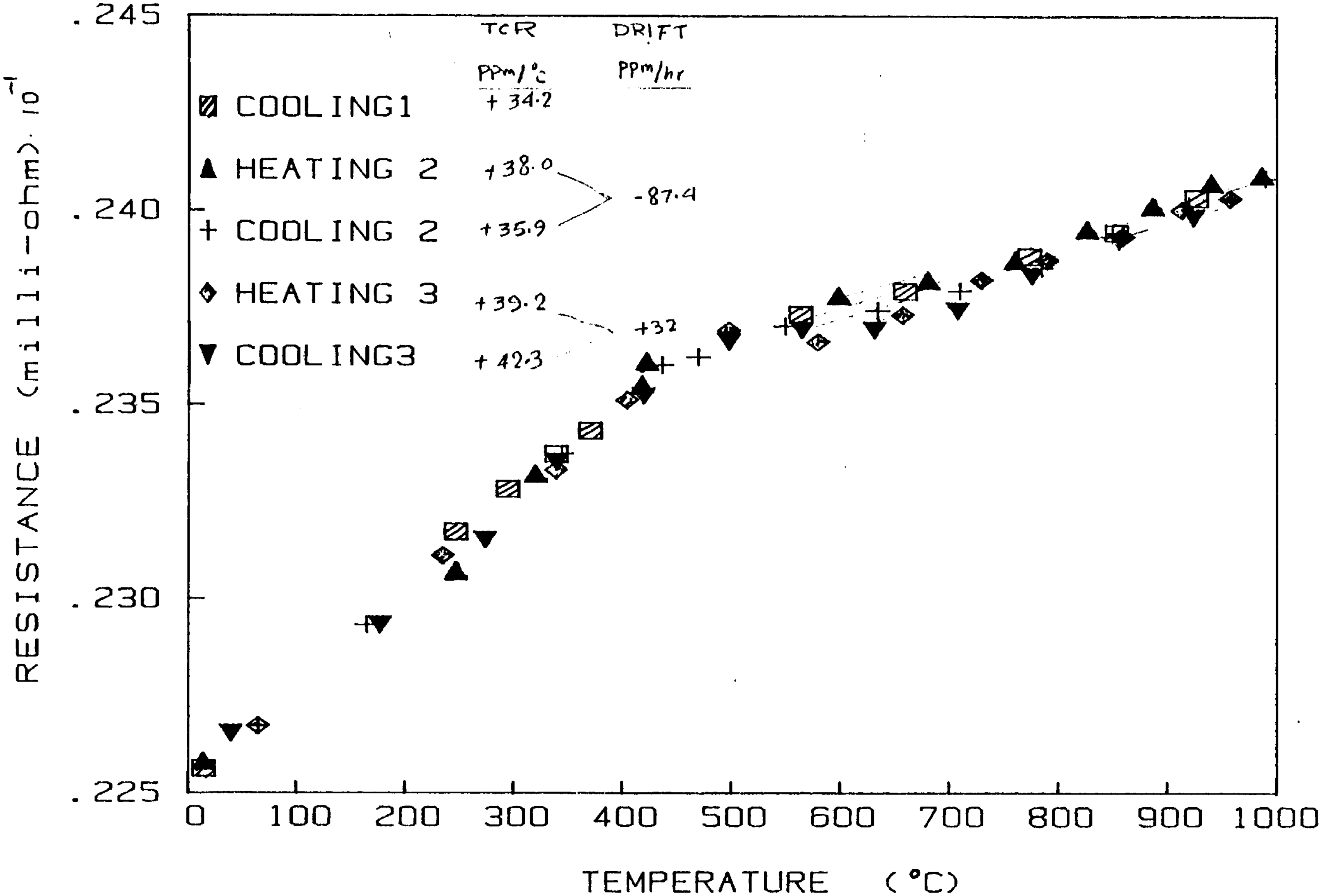
10 UNITS/DIVISION



Al-79.3 Wt.% V-4.25 Wt.% Mo-4.25 Wt.% Cr

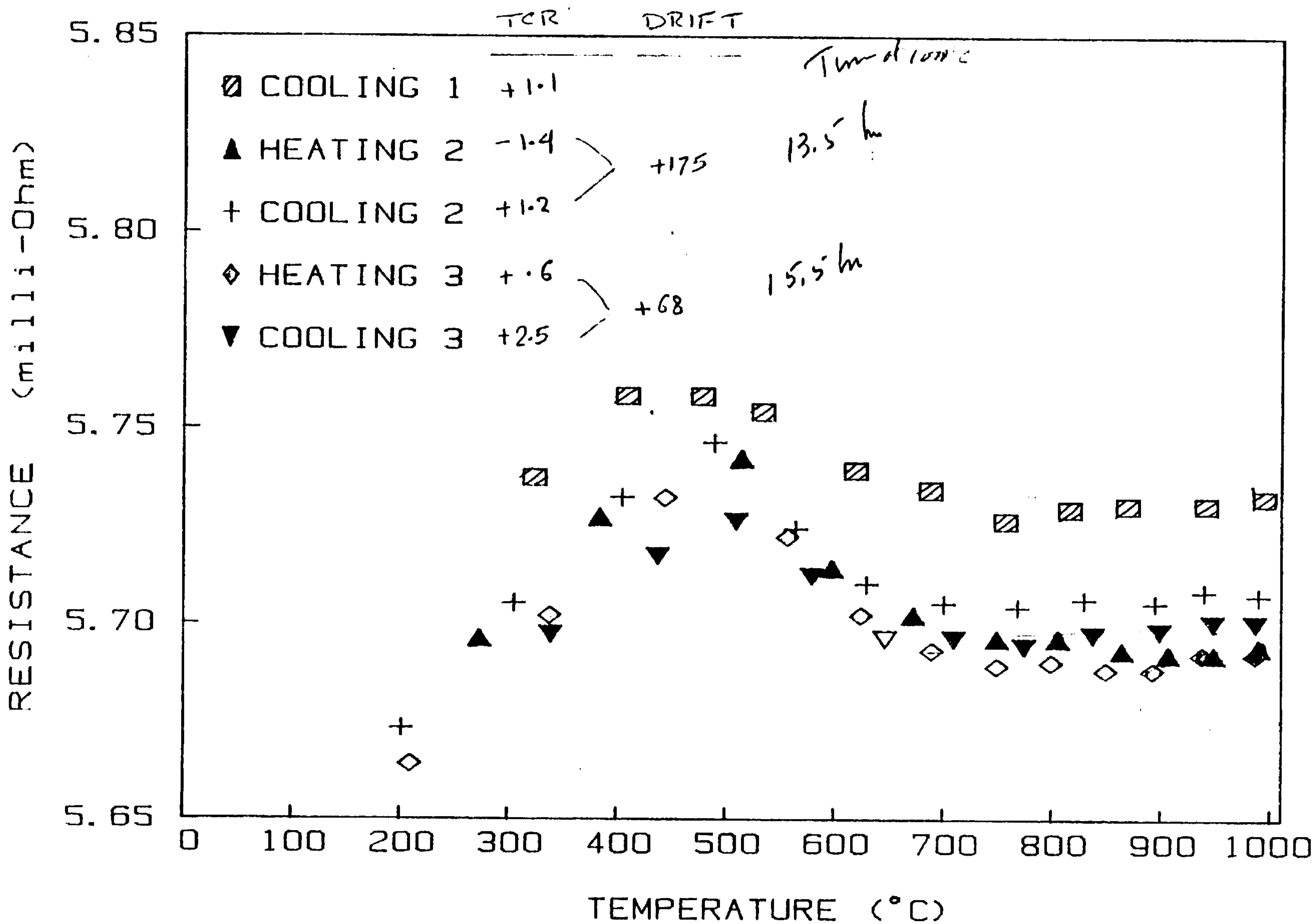
Figure 2A



Al-79.3 Wt.% V-4.25 Wt.% Mo-4.25 Wt.% Cr

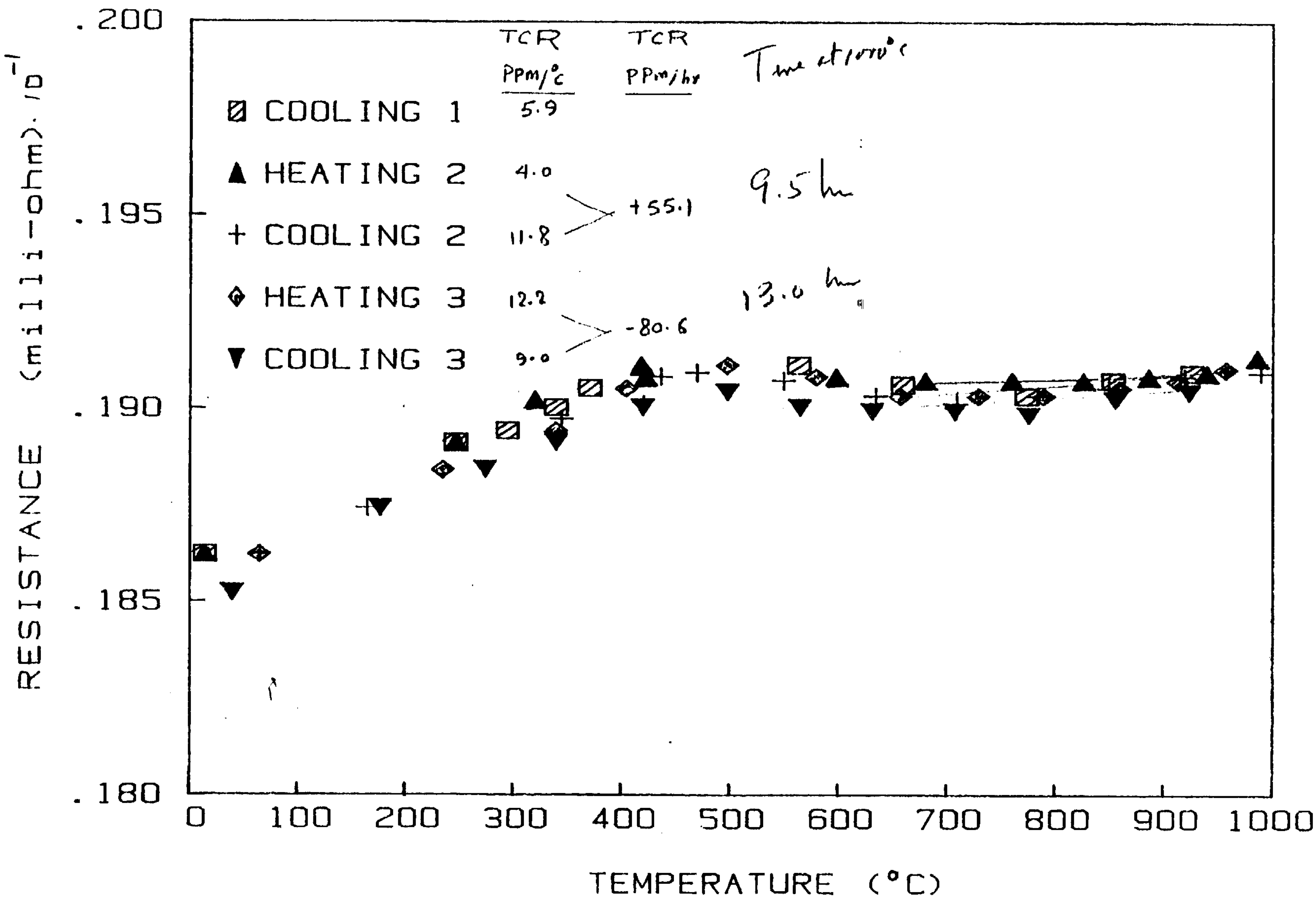
Prior Anneal 48 hrs at 1030°C

Figure 2B



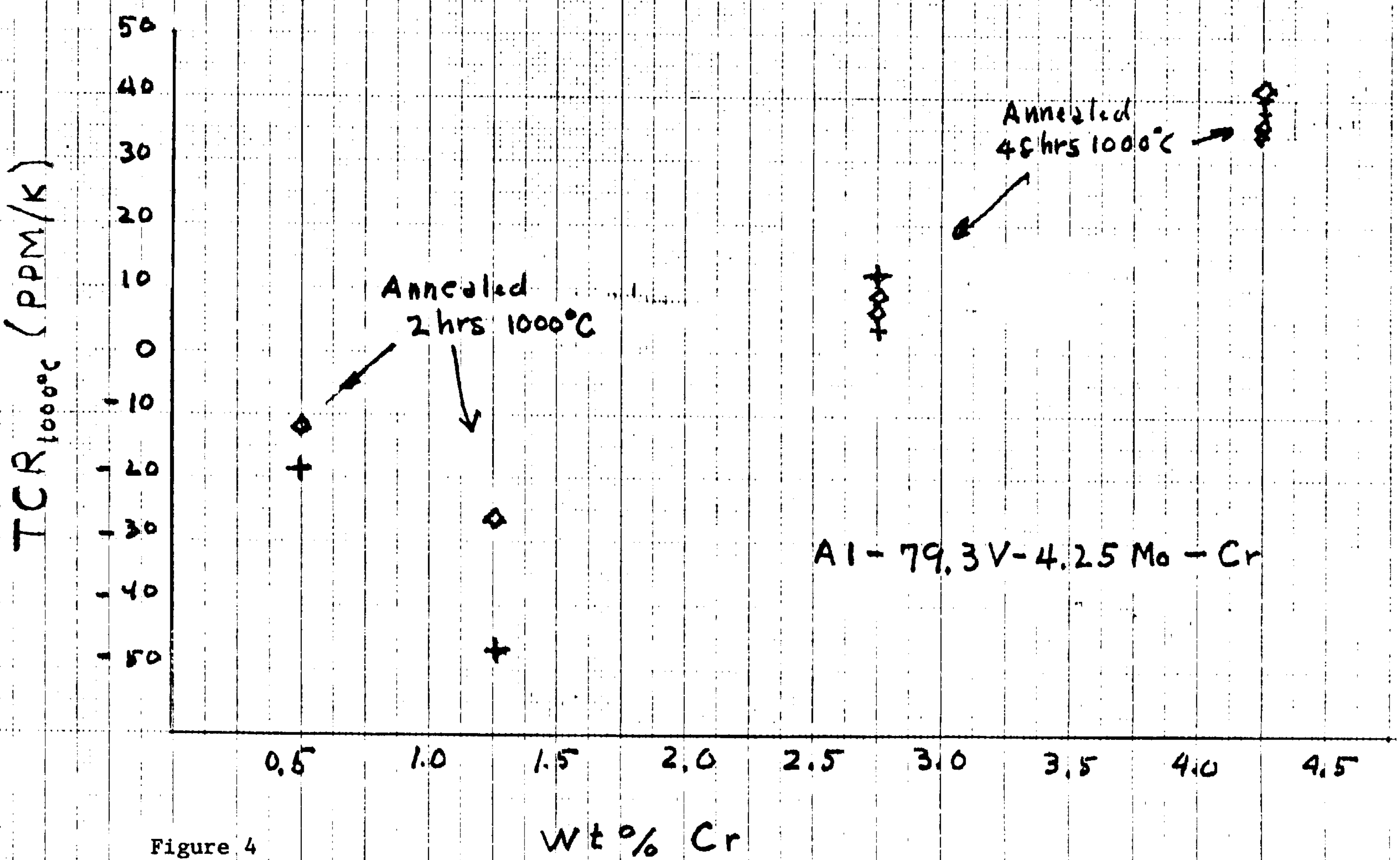
Al-79.3 Wt.% V-4.25 Wt.% Mo-2.75 Wt.% Cr

Figure 3A



Al-79.3 Wt.% V-4.25 Wt.% Mo-2.75 Wt.% Cr
Prior Anneal 48 hrs at 1030°c

Figure 3B



$TCR_{1000°C}$ (PPM/K)
50
40
30
20
10
0
-10
-20
-30
-40
-50
Annealed
2 hrs 1000°C
Annealed
48 hrs 1000°C
Al - 79.3 V - 4.25 Mo - Cr
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
Wt % Cr

Figure 4

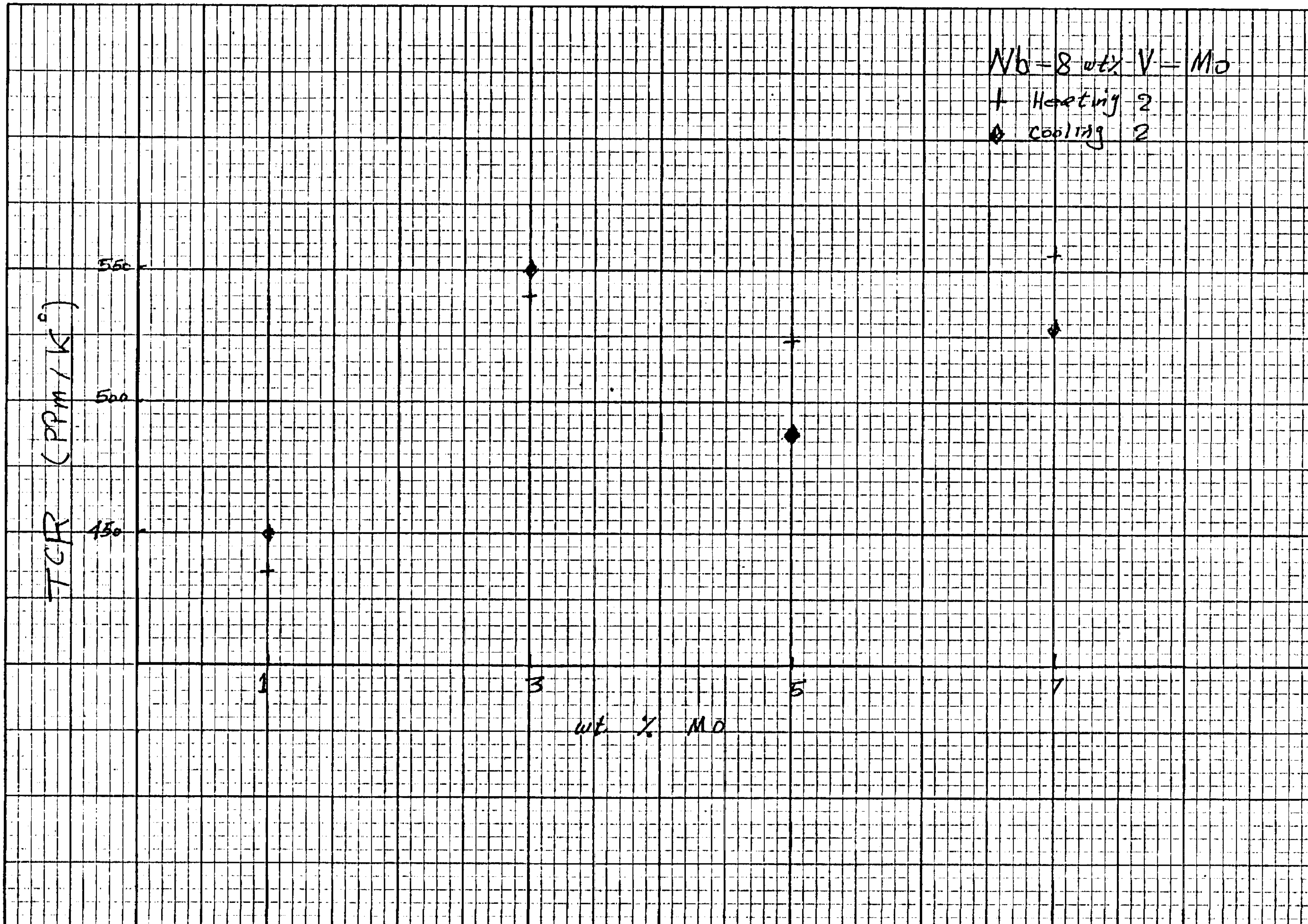


Figure 5

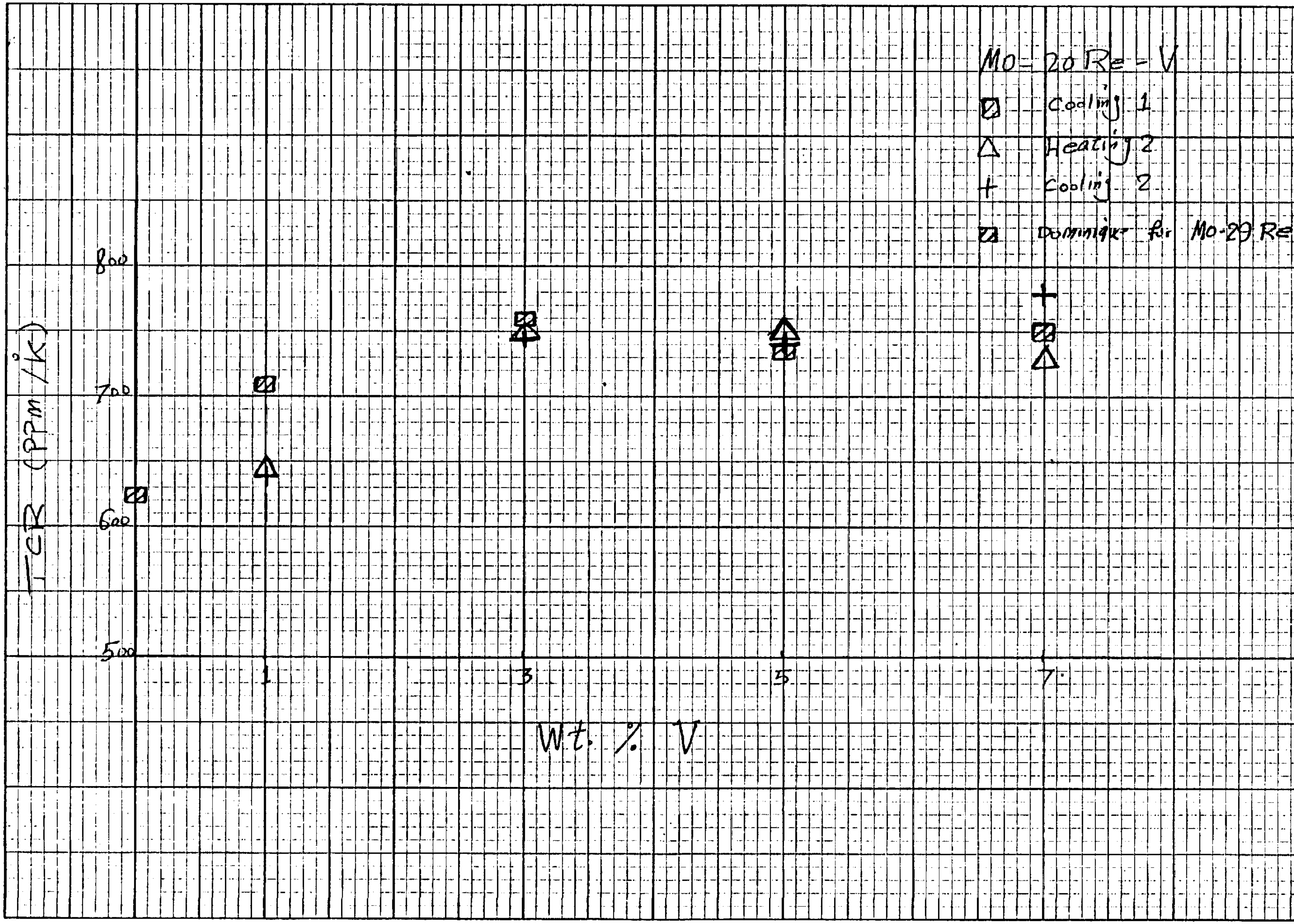


Figure 6

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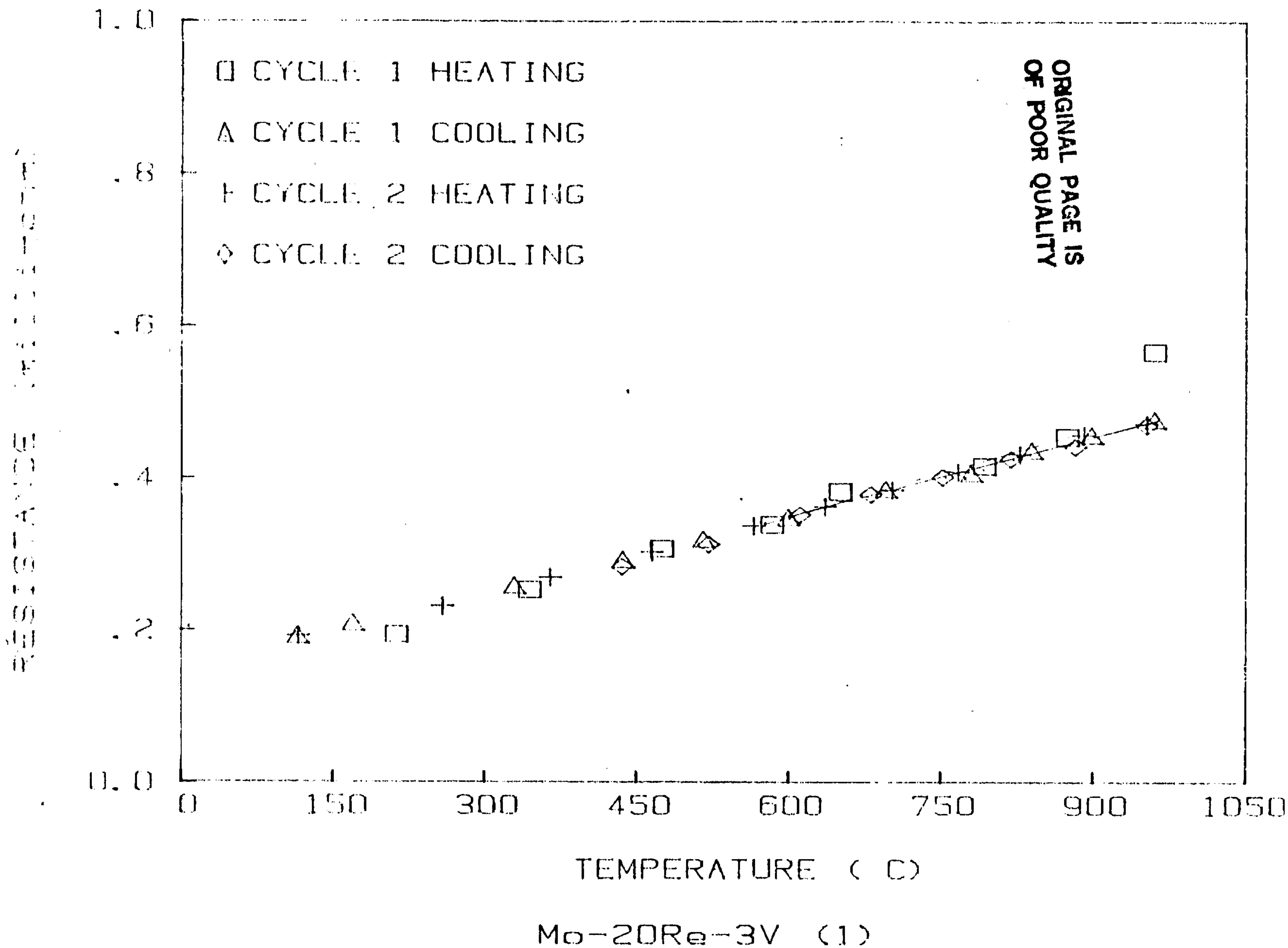

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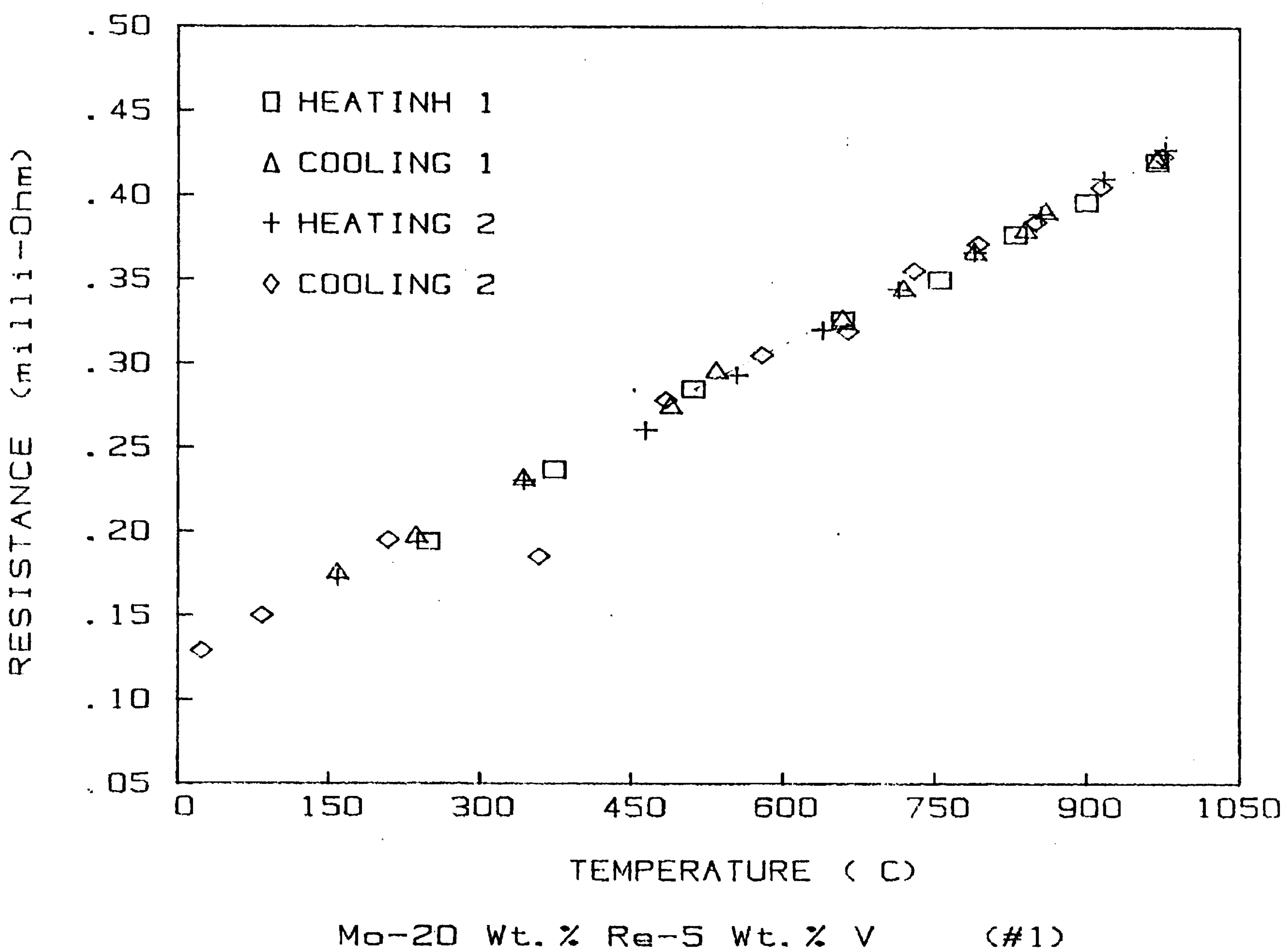
Figure 7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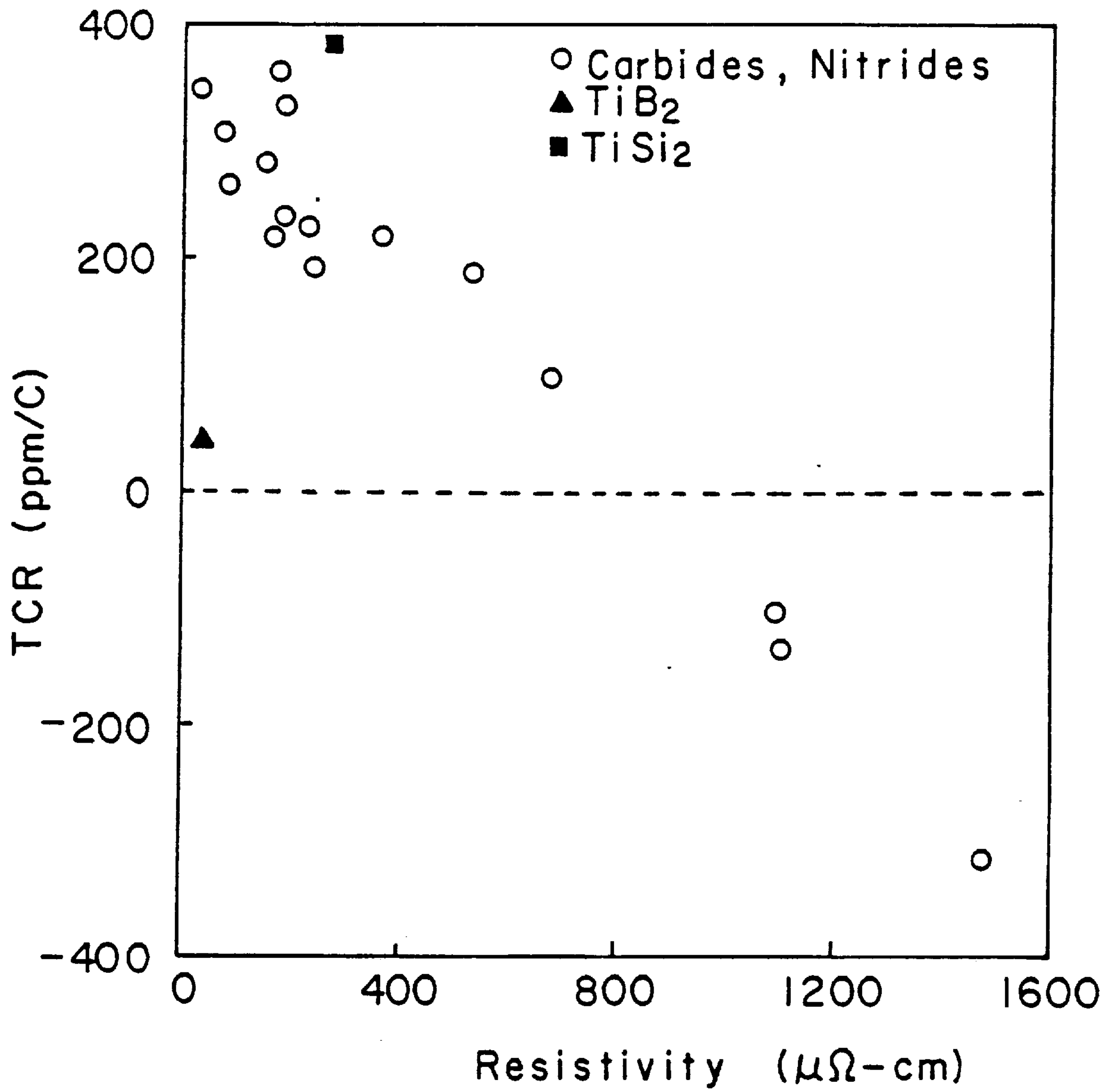
COOLING 1
HEATING 2
COOLING 2
.50
.45
.40
.35
.30
.25
.20
.15
.10
.05
RESISTANCE (milli-Ohm)
0
150
300
450
600
750
900
1050
TEMPERATURE (C)
Mo-20 Wt.% Re-5 Wt.% V (#1)

Figure 8



400
200
0
-200
-400
TCR (ppm/C)
0
400
800
1200
1600
Resistivity (μΩ-cm)
Carbides, Nitrides
TiB2
TiSi2

Figure 9

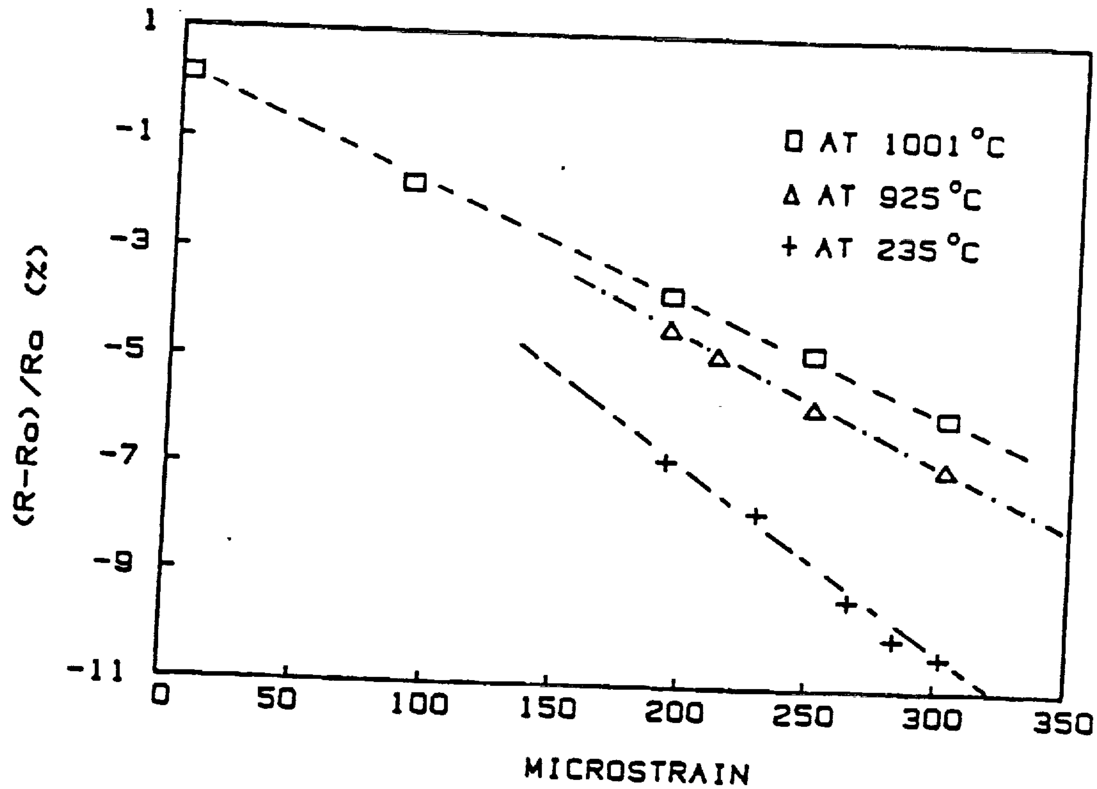
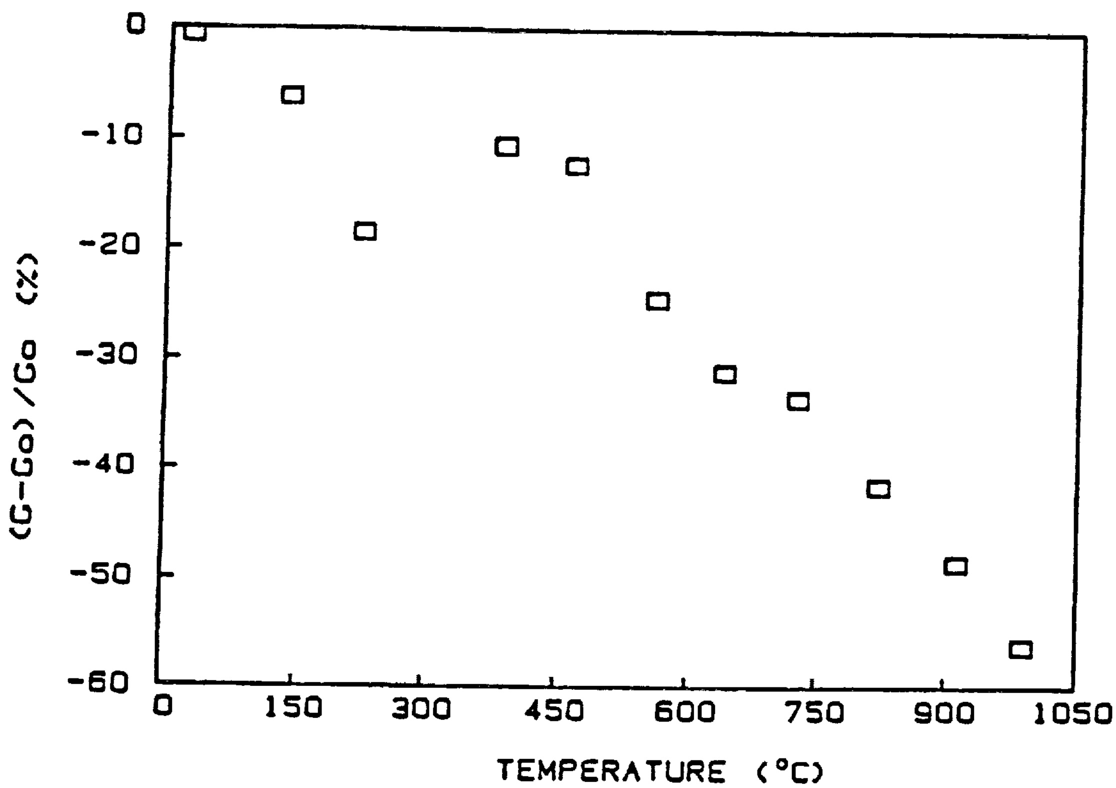

1
-1
-3
-5
-7
-9
-11
(R-Ro)/Ro (%)
0
50
100
150
200
250
300
350
MICROSTRAIN
□ AT 1001 °C
Δ AT 925 °C
+ AT 235 °C

Figure 10


0
-10
-20
-30
-40
-50
-60
(G-Go)/Go (%)
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150
300
450
600
750
900
1050
TEMPERATURE (°C)

Figure 11

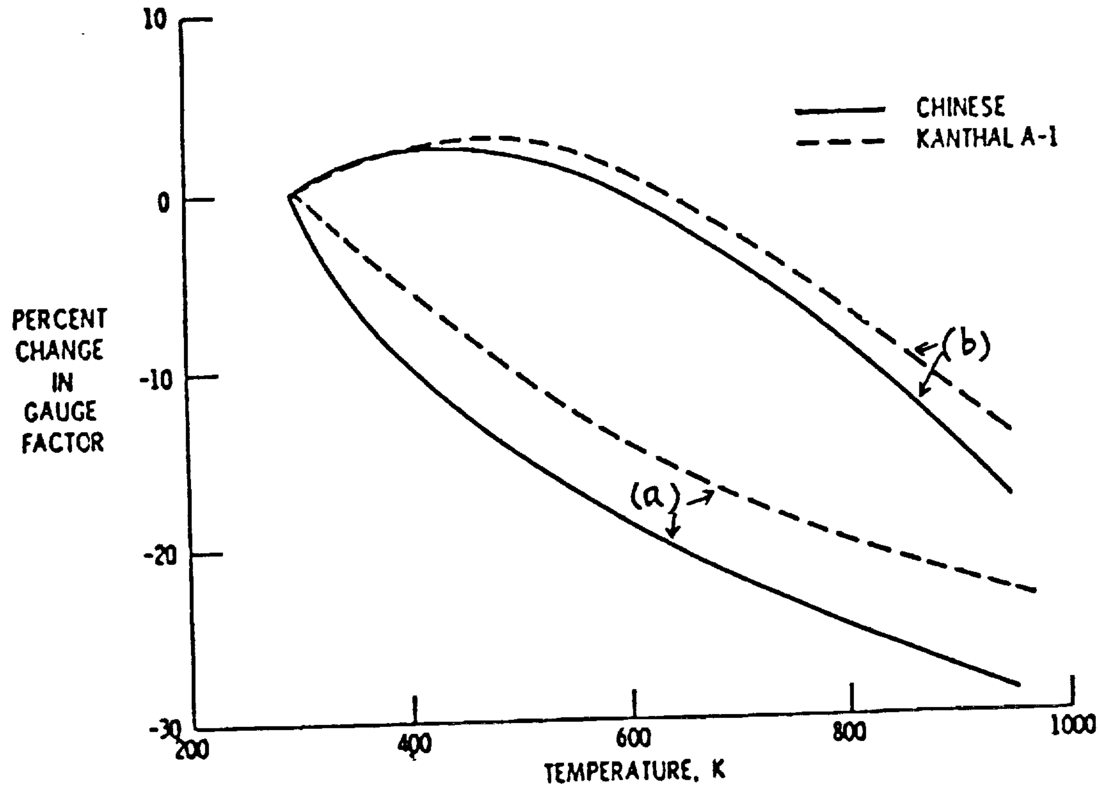
10
0
-10
-20
-30
PERCENT CHANGE IN GAUGE FACTOR
CHINESE
KANTHAL A-1
(a)
(b)
200
400
600
800
1000
TEMPERATURE, K

Figure 12