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Contents

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ABSTRACT

The kinetics of auto-tempering were studied in five steels--AISI 1090, AISI 1046, AISI 4340, AMS 6416 (300-M), and modified 4340+3%Si at six tempering temperatures, 100°C, 150°C, 180°C, 240°C, 270°C and 300°C by an electrical resistance method. With increasing isothermal tempering temperatures (100-300°C) the effects of the alloying elements, upon the tempering rate, became progressively greater. Silicon was observed to retard the tempering rate with increasing tempering time at a fixed temperature. Also, the ductility of steels tempered at 180°C was found to increase with tempering time and to decrease with increasing silicon content.

I. INTRODUCTION

Many articles have been written on the tempering of martensite in steels. The tempering process involves the segregation of carbon, the precipitation of carbides, the decomposition of retained austenite, and the recovery and recrystallization of the martensitic structure. Because some of these reactions overlap and occur on such a fine scale, the understanding of them is not complete. Even though the tempering processes of plain carbon steels have been thoroughly studied and are reasonably well understood, much more information is still needed to completely understand the complex effects of alloying elements upon the tempering treatment.[1]

In this investigation two plain carbon steels, AISI 1090 and AISI 1046, and three alloy steels, AISI 4340, AMS 6416 (300-M), and modified 4340+3%Si, were studied to determine the effects of alloying elements in general, and silicon in particular, upon the kinetics of auto-tempering. Specifically, this study was concerned with the first step of the tempering process, the redistribution of carbon to dislocations,[2] and with the so-called first and third stages of tempering, the precipitation of ϵ-carbide in low carbon martensite and the formation of cementite and ferrite.[3]

Auto-tempering (sometimes called quench-tempering) usually occurs during the transformation of steel from austenite to martensite.[4] Generally speaking, it occurs during the brief period available for carbon atoms to redistribute themselves during the quenching process. In thick specimens, auto-tempering occurs as a result of the inside of the sample cooling at a slower rate than the outside, hence the steel actually tempers during the quench. To study the kinetics of auto-tempering, it is necessary to suppress any tempering during the quench. This can be accomplished by very rapid quenching. However, the "so-obtained" martensite is often mixed with small amounts of retained austenite, which can be minimized by subsequent cooling to liquid nitrogen temperatures. The fast cooling can be accomplished by using a small diameter wire. With wire specimens, electrical resistance changes provide an excellent means for monitoring the initial kinetics of the tempering process.

The electrical resistance method was selected from a variety of possible experimental techniques (X-ray diffraction,[5-8] dilatometry,[3,5] hardness,[9] magnetic,[10] and electrical resistivity[11,12]) that have been used to study the kinetics of first stage tempering in plain carbon steels. This method was chosen because the resistance change, which is a sensitive function of the tempering reaction, can be easily and accurately measured with wire specimens almost immediately because the wire coil attains the tempering bath temperature within 2 secs. The other techniques were considered but eliminated for various reasons. For example, X-ray diffraction was eliminated because of the problems of line broadening and strain relief.[6] Use of the dilatometry technique is restricted by the sample size and instrument instability at the beginning of the reaction.[5]

Originally, _in situ_ measurements in the tempering bath were to be conducted, but because of serious heating problems and difficulties in obtaining an _in situ_ high temperature tempering medium, the specimens were tempered in the bath and the resistance then measured at room temperature. The experimental method was successful and provided the desired tempering data.

A secondary objective of this investigation was to study the influence of tempering on the ductility.[13] A measure of ductility was obtained by stretching the coiled specimens at room temperature until they broke.

II. EXPERIMENTAL PROCEDURES

A. Material Preparation

The 1090 steel used in this study was obtained commercially in wire form with the desired diameter of 0.020 in. (0.051 cm). The 1046 steel was received commercially as a 1/4 in. diameter rod and then was cold-swaged to 0.020 in. diameter wire. The alloy steels, AISI 4340 and AMS 6416 (300-M) were received as 5/8 in. thick bar stock in the fully annealed condition. The 4340+3%Si steel was made from commercial AISI 4340 steel that was remelted and to which 3%Si was added. A 4 lb. ingot was homogenized in vacuum for 24 hrs at 1200°C and then forged and hot rolled into bar stock, 2 1/2 in. wide and 5/8 in. thick. Finally, the bar was softened by holding it at 600°C for 4 hrs, leaving it in the same soft condition as the 4340 and 300-M. Sections 3 in. long were then cut from bars of each of the three alloy steels. These pieces were hot rolled at 900°C to 1/8 in. thick and then sheared into 1/8 in. wide strips. Next, the strips were wire drawn down to 0.020 in. diameter wire, with annealing being done when necessary.

Finally, pieces 8 in. (20.3 cm) in length were cut from the wires of all five steels and wound into coils 1-1/4 in. in length. The exact dimensions of the specimens are given in Fig. 4B. The chemical compositions of the steels are listed in Table I.

B. Transformation Technique and Apparatus

Before heat treating, 6 in. long chromel leads were resistance welded to the ends of each specimen, as shown in Fig. 4. The leads were later used to measure the resistance changes during tempering. The specimen was then attached to the sample-holder and austenitized at about 910°C in a helium (99.998% pure) atmosphere. The austenitization was carried out by passing current through the coil sample for 2 min. The approximate austenitizing temperature was measured by an optical pyrometer. (The austenitizing time and temperature were determined and the absence of specimen oxidation was confirmed, on the basis of various preliminary tests and subsequent chemical analysis.) Specimens were quenched by simultaneously shutting the current off and pushing the sample-tube assembly into a 10% brine bath. (The quenching rate was sufficiently high so that auto-tempering was minimized. All the steels, except 1090 transformed nearly completely to martensite, although small amounts of retained austenite were undoubtedly present in some of the steels.) After quenching, all specimens were immediately transferred to the liquid nitrogen storage chamber where they were held until needed. The liquid nitrogen helped to insure transformation of the retained austenite.[15]

A schematic drawing of the transformation apparatus is provided in Fig. 1A, and a schematic drawing of the sample holder is displayed in Fig. 4A. A photograph of the equipment is shown in Fig. 2.

C. Tempering and Electrical Resistance Techniques and Apparatus

The tempering studies were carried out isothermally at six temperatures, 100°C, 150°C, 180°C, 240°C, 270°C and 300°C. Tempering data at the higher temperatures (240°C, 270°C, 300°C) were collected over 3 hour periods, while data for specimens tempered at the lower temperatures (100°C, 150°C, 180°C) were obtained for 100 seconds, with the exception of the 100°C data which was collected for 1 hour. Boiling water was used for 100°C tempering; a thermostatically controlled salt pot (±3/4°C) was used for the other temperatures.

The changes in electrical resistance during tempering were determined as a function of time, with all measurements being made at room temperature. Since fluctuations in room temperature affect the electrical resistance, standards were used to calibrate the system for room temperature changes. The standard specimen of each steel was identical to the test specimen in every way except that it was quenched and tempered at 345°C in a salt bath for 25 hrs. A variation of 1° in room temperature causes the resistance to change by about 0.20%. A current of 0.1A was passed through the sample circuit from a constant current source. The two chromel leads spot-welded to the ends of the sample were connected to a differential voltmeter to measure the potential drop across the coil. Voltmeter readings were reproducible within ±0.075%. A schematic drawing of the resistance measuring apparatus is provided in Fig. 1B. A photograph of the tempering equipment is shown in Fig. 3, and a close-up of the sample-holder is provided in Fig. 5. Figure 6 displays the block diagram for the electrical circuit.

The experimental procedure for making the electrical resistance measurements consisted of the following steps:

1. The voltmeter and constant current source were turned on and remained on throughout the entire investigation; current was passed through the specimen only when measurements were being made.

2. The resistance of the standards was measured and the system calibrated. (The standard was measured before and after the completed tempering test of each specimen.)

3. The specimen was removed from the liquid nitrogen container and dried with a blast of compressed air.

4. The specimen was then attached to the stainless steel terminals, the leads were connected to the voltmeter, and the current was turned on.

5. The resistance of the sample was measured, and the room temperature was recorded to a 1/4°C.

6. The current was turned off, and the coil specimen was removed from the holder and placed in the tempering bath.

7. The specimen was tempered for the selected time, with time being allowed for the sample to reach the bath temperature. (Sample warm-up time was determined by X-Y recorder tests. Specimens tempered in boiling water reached 100°C in a fraction of a second; 1 sec was allowed for the specimens to reach 180°C and above, while 2 secs were allowed for the specimens to attain the 150°C tempering temperature.)

8. After tempering, the specimen was immediately quenched in water, removed and then dried with a blast of air.

9. The specimen was attached again to the specimen holder and the resistance measured.

10. The procedure from step 4 was repeated continuously for the duration of the particular tempering study.

D. Ductility Test

A ductility test was conducted on specimens of all the steels, except 1090, in the "as-quenched" condition and after tempering at 180°C for 2 secs, 10 secs and 100 secs. The test was also performed on the coils of all steels tempered at 100°C for 1 hr.

The test consisted of manually pulling the coil specimen apart until it broke. By visually comparing the resulting changes in length of the broken specimens, a measure of relative ductility was established.

III. RESULTS AND DISCUSSION

A. General Discussion

The results of this resistometric study are expressed as the ratio, ($\Delta R/R$) of the change in resistance to the initial resistance of the steel measured at room temperature in "as-quenched" condition. Throughout this investigation, the estimated error in making each measurement is indicated by the circle size on the graphs.

To correlate the present obtained data with that of earlier studies, the initial resistance readings for all five steels have been converted to electrical resistivities, which are listed in Table II. The initial resistivities for the 1090 and 1046 steels are slightly lower than those reported by previous investigators.[2,11]

The rate of change of resistance was found to vary with carbon content in the plain carbon steels and with silicon content in the alloy steels. The magnitude of the resistance changes at a fixed time increased sharply with tempering temperature, and it was on this basis that the results have been grouped together. The low temperature studies consisted of the steel specimens tempered at 100°C, 150°C and 180°C, while the high temperature studies consisted of those samples tempered at 240°C, 270°C and 300°C.

B. Low Temperature Studies

During the tempering of the steels at the low temperatures, the initial step of tempering occurs[2] and the so-called first stage of tempering begins and becomes almost complete in the plain carbon steels.[5,17] The resistance changes for all five steels tempered at the three temperatures are tabulated in Table III, and the resulting curves are shown in Figs. 7-11.

In all five steels the effect of tempering was to cause a progressive decrease in the electrical resistance with tempering time. In agreement with the known kinetics of the so-called first stage of tempering, the reaction was observed to start without an incubation period and proceed at a rapidly diminishing rate.[2,5,11] The magnitude of the resistance changes in plain carbon steels increased greatly with carbon content, as illustrated by the 1090 and 1046 curves, while the magnitude of the changes in the alloy steels increased with decreasing silicon content, as indicated by comparing the 4340, 300-M, and 4340+3%Si curves at all three temperatures.

At 100°C, the steels were tempered for the first 100 secs to observe the auto-tempering rate and then tempered for a total of 1 hr. to determine the effect of a longer tempering time. The curves for all five steels show that the resistance change initially decreases at a gradual rate during the first 100 secs. During the remaining 1 hr of tempering, however, there is a relatively large resistance decrease. This result is different from that obtained for the steels tempered at 150°C and 180°C, where much of the tempering occurs in the first few seconds and then the rate gradually diminishes with time. These characteristics at 100°C also have been observed by King and Glover.[11,18]

Tempering at temperatures of 150°C and 180°C resulted in curves for all five steels which showed the electrical resistance decreasing continuously with time. There was no initial "gradual rate change" region like that found at 100°C. Presumably, carbide precipitation begins almost immediately upon tempering at these temperatures and the resistance decreases continuously as this precipitation continues. This behavior is consistent with the formation of ε-carbide.[2]

In order to get a better indication of alloying element effects, and in particular the influence of silicon, the absolute resistance changes with tempering time at these temperatures were determined and plotted. The values are listed in Table IV and the curves are shown in Fig. 12 for the 1046, 4340, 300-M and 4340+3%Si steels.

At 100°C, within the first 100 secs of tempering, the absolute resistance changes are practically the same for all four steels. After 100 secs, however, and up to 1 hr the resistance differences among the steels increase rapidly with time. It appears that within the first 100 secs the alloying elements have very little, if any, effect on the tempering rate. After the first few minutes, however, the alloying elements, particularly silicon, exert sufficient influence to decrease the tempering rate. As a result, after 1 hr there is a relative difference among the resistance changes such that 1046 $\gg$ 4340 $>$ 300-M $>$ 4340 $>$ 4340+3%Si, as indicated in Fig. 12. At 150°C and 180°C, the differences in absolute resistance change among the steels occur almost immediately and become greater with time. The rate changes between the higher silicon steels (300-M and 4340+3%), however, remain relatively the same throughout the first 100 secs of tempering at both temperatures. Thus, at 150°C and 180°C the alloying element effect (particularly silicon) is immediately apparent, whereas, at 100°C the alloying element effect is not really noticeable until after at least 100 secs of tempering. The results found here are contrary to earlier studies,[17,19] which stated that silicon does not alter the kinetics of first stage tempering, but are in agreement with the results of King and Glover, who found silicon to retard the first stage of tempering in steels.[18]

An attempt was made to calculate activation energies for carbon diffusion in the five steels at the three lower tempering temperatures. The values were determined in the customary manner from the rate equation:

$$\frac{1}{t} = A \exp(-Q/RT)$$

where t is time, A is a constant, R is the gas constant, and T is the reaction temperature in absolute degrees.[20] The empirical activation energy, Q, was estimated by solving the equation simultaneously (for Q) at two temperatures with the corresponding time necessary to reach a certain stage of the reaction. A sample calculation is shown in the Appendix. The subsequent activation energy values are an average of the results determined from combinations of the three temperatures.

Because of the large resistances changes that occur so rapidly, accurate estimates of activation energy cannot be obtained at the start of the reaction, consequently, the activation energies were calculated after at least 10 seconds of tempering. The calculated activation energies were found to increase with tempering time at the beginning of the reaction. The range of the activation energy for each of the steels is listed in Table V.

It appears that in both plain carbon steels the first stage reactions occur relatively quickly, as indicated by the narrow activation energy range, and they approach a steady state. In the alloy steels, the process occurs at slower rates so that longer tempering times are necessary to complete the reaction. The differences in activation energies between the plain carbon and alloy steels also indicate the effect of silicon in retarding tempering. The activation energies estimated for the 1090 and 1046 steels are in reasonable agreement with earlier reported values.[5,21] The result of activation energies increasing with reaction time at the beginning of the tempering process was theorized by Hillert,[22] and has been established experimentally by Gerdien.[23]

C. High Temperature Studies

The resistance change-time curves of specimens tempered at 240°C, 270°C, and 300°C were similar to those tempered at 150°C and 180°C. The magnitude of the resistance changes, however, was much greater and increased to a greater degree with increases in tempering temperature. In all five steels, a large part of the resistance change occurs within the first five seconds, after which, the rate of electrical resistance changes continuously decreases with time at all temperatures. The resistance changes for the specimens tempered at the higher temperatures are listed on Tables VI through VII and the resulting curves are shown in Figs. 13-17.

In both of the plain carbon steels, a large amount of tempering occurs within the first 5 secs, yet a significant part still occurs within the next 3 hrs. This trend is probably a result of the so-called third stage of tempering, i.e., the formation of cementite and ferrite which occurs within this tempering range.[2,24] The magnitude of the resistance changes for the 1090 steel is much greater than the changes for the 1046 steel, as is to be expected.

In the alloy steels, the 4340 steel tempers at a faster rate than the 300-M and the 300-M tempers faster than 4340+3%Si. This can be seen by comparing the slopes of the curves after the first few seconds of tempering. The data clearly show that the addition of silicon retards the tempering reactions occurring at these temperatures. This silicon effect has been reported by earlier investigators.[17,19,25,26]

The "apparent" activation energies of each of the steels for the complex reactions occurring at these tempering temperatures are listed in Table IX. The activation energies were found to increase with initial tempering time, but to a lesser extend than those calculated at the lower temperatures; consequently, only the average estimates are tabulated. The activation energies were calculated by the method described previously in determining the lower temperature results.

The "apparent activation" energies calculated for the 1090 and 1046 steels at these high temperatures are somewhat near their calculated values for the first stage of tempering at the lower temperatures. The values calculated for the alloy steels are much higher than those of 1090 and 1046, and are definitely influenced by the silicon content.

Increasing the silicon in the steel increases the "apparent" activation energy for the tempering reactions at these temperatures. Similar activation energy values were reported by Owen in his investigation of silicon effects on the kinetics of tempering for the third stage reactions.[19] The general effect of silicon on the kinetics of the tempering process at these temperatures is to retard the precipitation of cementite.[17,19,26]

D. Ductility Tests

Relative ductility tests were performed on steel specimens of 1046, 4340, 300-M and 4340+3%Si in the "as-quenched" condition and after tempering at 180°C for 2 seconds, 10 seconds and 100 seconds. The results are shown in the series of photographs as depicted in Figs. 18-21.

The steel specimens of 1046, 300-M and 4340+3%Si were all brittle in the "as-quenched" condition. However, the 4340 coil, did rather unexpectedly exhibit some ductility.

The relative ductility increased with tempering time for each of the steels tempered at 180°C. The magnitude of the ductility increase was inversely related to the silicon content of the steel. Thus with tempering time, the relative increase in ductility was much greater in the 1046 and 4340 steels than in the high silicon steels (300-M and 4340+3%Si). Generally, both the 1046 and 4340 steels showed relatively moderate increases in ductility with tempering time before breaking. The 300-M steel exhibited a ductility increase, but to a much lesser extent than the 1046 or 4340 steel. The 4340+3%Si steel showed a very small increase in ductility as a function of time, even after 100 secs of tempering. The contrast in ductility between the 1046 and 4340+3%Si steels after 100 secs of tempering was quite impressive, with the change in length of steel 1046 increasing by a factor of 1.5, whereas the 4340+3%Si length change increased by only 0.13.

The relative ductilities of the four steels tempered for 1 hour at 100°C is illustrated in Fig. 22. The steel ductility decreases with increasing silicon content, analogously to the 180°C tests. Thus, the order of ductility for the steels is 1046 > 4340 > 300-M > 4340+3%Si. The ductility of each steel tempered at 100°C also correlates reasonably to those specimens tempered at 180°C, when time and temperature are taken into account according to the rate equation. For example, the length of the pulled specimen of 4340 tempered at 100°C for 1 hr is between the length of those specimens tempered at 180°C for 2 secs and 10 secs.

IV. CONCLUSIONS

1. At the 100°C tempering temperature, the alloying elements were found to have very little effect upon the initial kinetics of tempering, but with longer tempering times the effect of silicon on retarding the tempering process was clearly evident.

2. With increasing tempering temperatures, the rate of tempering became greater, and with increasing tempering time the alloying element effects, especially that of silicon,became more dominant in reducing the tempering rate.

3. The activation energies increased with increasing tempering time for each steel; until a steady state was reached.

4. Ductility in the wire specimens tempered at 180°C was found to increase with increases in tempering times while the ductility decreased with increased silicon content.

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APPENDIX

Formula: $$\frac{1}{t} = A \exp(-Q/RT)$$

Simultaneous Equations:

$$\ln \frac{1}{t_1} = \ln A - Q/RT_1$$

$$\ln \frac{1}{t_2} = \ln A - Q/RT_2$$

Solving for Q: $$Q = \frac{R(\ln \frac{1}{t_1} - \ln \frac{1}{t_2})}{(\frac{1}{T_2} - \frac{1}{T_1})}$$

Example

For 1090 steel, corresponding to the stage of the process where $\Delta R/R = -8\%$

Reaction data: $t_1 = 36$ secs $T_1 = 423°K$ (150°C)

$t_2 = 3600$ secs $T_2 = 373°K$ (100°C)

$$R = 1.98 \text{ cal-°C/mole}$$

Substituting:

$$Q = (1.98) \frac{(\ln \frac{1}{36} - \ln \frac{1}{3600})}{(\frac{1}{373} - \frac{1}{423})}$$

$$Q = \frac{9.12}{(\frac{1}{373} - \frac{1}{423})}$$

$$Q = 28{,}800 \text{ cal/mole}$$

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Table I. Chemical composition of steels (given in wt%).

Steel	C	Cr	Mn	Mo	Ni	Si	V
AISI 1090	0.90	0.05	0.79	<0.01	0.05	0.23	--
AISI 1046	0.46	0.04	0.68	<0.01	0.04	0.16	--
AISI 4340	0.40	0.74	0.71	0.23	1.72	0.28	--
AMS 6416 (300-M)	0.39	0.79	0.73	0.41	1.72	1.64	0.06
4340 + 3% Si	0.40	0.72	0.70	0.22	1.66	3.23	--

Table II. Initial electrical resistivity.

Steel	ρ (μohm-cm)
1046	23
1090	28
4340	33
300-M	52
4340 + 3% Si	65

Table III. Resistance changes during isothermal tempering at 100°C, 150°C and 180°C.

ΔR/R%, After Indicated Times

	Steel				
Time	1090	1046	4340	300-M	4340 + 3%Si
at 100°C					
2 sec	0.60	0.25	0.10	0.06	0.07
5 sec	0.87	0.45	0.17	0.10	0.10
10 sec	1.15	0.55	0.27	0.15	0.13
30 sec	1.75	0.70	0.45	0.21	0.17
100 sec	2.35	0.95	0.62	0.31	0.23
3600 sec	8.00	3.69	1.92	0.89	0.57
at 150°C					
2 sec	3.24	1.47	0.91	0.53	0.40
5 sec	5.00	2.18	1.31	0.79	0.57
10 sec	6.11	2.80	1.68	1.00	0.72
30 sec	7.69	3.50	2.28	1.30	0.92
100 sec	9.58	4.41	2.75	1.55	1.21
at 180°C					
2 sec	6.57	2.76	1.70	0.94	0.70
5 sec	8.49	3.73	2.35	1.27	0.97
10 sec	9.57	4.36	2.75	1.49	1.14
30 sec	11.40	5.14	3.23	1.76	1.35
100 sec	12.66	5.87	3.74	2.04	1.59

Table IV. Absolute resistance changes during isothermal tempering at 100°C, 150°C and 180°C.

	ΔR ($\times 10^{-3}$ ohms) after Indicated Times			
	Steel			
Time	1046	4340	300-M	4340 + 3% Si
at 100°C				
2 sec	0.5	0.3	0.3	0.4
5 sec	0.9	0.5	0.5	0.6
10 sec	1.1	0.8	0.7	0.8
30 sec	1.4	1.3	1.0	1.0
100 sec	1.9	1.8	1.5	1.4
3600 sec	7.4	5.6	4.3	3.4
at 150°C				
2 sec	3.1	2.7	2.5	2.4
5 sec	4.6	3.9	3.7	3.4
10 sec	5.9	5.0	4.7	4.3
30 sec	7.4	6.8	6.1	5.5
100 sec	9.3	8.2	7.3	7.2
at 180°C				
2 sec	5.7	5.0	4.3	4.1
5 sec	7.7	6.9	5.8	5.7
10 sec	9.0	8.1	6.8	6.7
30 sec	10.6	9.5	8.0	7.9
100 sec	12.1	11.0	9.3	9.3

Table V. Values of Q at Low Temperatures. (100°,150°,180°C) Q, cal/mole.

Steel	Range
1090	28,800-29,100
1046	27,500-28,100
4340	30,000-33,900
300-M	31,800-38,100
4340 + 3% Si	32,600-41,100

Table VI. Resistance changes during isothermal tempering at 240°C.

Time (sec)	ΔR/R%, after Indicated Times — Steel 1090	1046	4340	300-M	4340 + 3%Si
3	17.45	5.81	4.11	2.49	1.84
5	18.06	6.36	4.41	2.65	1.93
10	18.80	7.00	4.68	2.82	1.99
20	19.69	7.54	4.91	2.96	2.08
30	20.18	7.79	5.04	3.02	2.13
60	21.16	8.28	5.31	3.16	2.20
100	21.89	8.63	5.47	3.25	2.26
120	22.13	8.73	5.57	3.29	2.30
180	22.66	8.88	5.80	3.36	2.33
300	23.35	9.22	6.04	3.49	2.40
600	24.17	9.58	6.30	3.63	2.50
900	24.49	9.81	6.47	3.69	2.53
1,200	24.82	9.91	6.60	3.76	2.60
1,800	25.22	10.11	6.73	3.87	2.64
6,000	25.87	10.55	7.20	4.14	2.79
9,600	26.12	10.75	7.36	4.29	2.84
12,000	26.20	10.80	7.46	4.34	2.86

Table VII. Resistance changes during isothermal tempering at 270°C.

Time (sec)	ΔR/R%, after Indicated Times				
	Steel				
	1090	1046	4340	300-M	4340 + 3%Si
3	18.79	6.84	4.70	3.10	2.17
5	19.61	7.51	4.99	3.27	2.25
8	20.20	7.95	5.15	3.37	2.30
14	21.02	8.34	5.37	3.49	2.36
25	21.80	8.67	5.59	3.58	2.44
36	22.39	8.92	5.71	3.69	2.49
66	22.97	9.35	6.06	3.82	2.58
108	23.48	9.54	6.22	3.90	2.63
130	23.68	9.64	6.28	3.94	2.64
190	24.15	9.69	6.44	4.00	2.68
310	24.62	9.98	6.63	4.09	2.75
620	25.40	10.31	6.94	4.23	2.88
900	25.60	10.46	7.13	4.36	2.95
1,200	25.87	10.65	7.29	4.41	2.98
1,800	26.18	10.75	7.48	4.49	3.00
6,000	26.81	11.18	7.86	4.70	3.14
9,600	26.97	11.33	7.95	4.81	3.17
12,000	27.05	11.42	7.99	4.83	3.19

Table VIII. Resistance changes during isothermal tempering at 300°C.

Time (sec)	ΔR/R%, after Indicated Times — Steel: 1090	1046	4340	300-M	4340 + 3%Si
3	20.54	7.76	5.55	3.65	2.61
5	21.32	8.28	5.79	3.78	2.65
8	22.01	8.70	5.97	3.89	2.72
14	22.75	9.13	6.19	4.01	2.80
25	23.40	9.47	6.50	4.16	2.84
36	23.98	9.70	6.63	4.23	2.89
66	24.63	9.99	6.90	4.33	2.98
108	25.08	10.27	7.10	4.42	3.02
130	25.20	10.32	7.20	4.44	3.04
190	25.49	10.46	7.30	4.51	3.07
310	25.82	10.70	7.54	4.61	3.12
620	26.23	10.88	7.64	4.70	3.21
900	26.55	11.03	7.74	4.78	3.25
1,200	26.72	11.07	7.84	4.85	3.28
1,800	26.96	11.17	7.98	4.93	3.33
6,000	27.62	11.59	8.25	5.10	3.43
9,600	27.70	11.69	8.31	5.15	3.46
12,000	27.78	11.74	8.38	5.19	3.49

Table IX. Values of ΔH at high temperatures. (240°,270°,300°C)

Steel	ΔH, cal/mole (average)
1090	27,700
1046	29,600
4340	34,600
300-M	51,300
4340 + 3% Si	60,000

FIGURE CAPTIONS

Fig. 1. (A) Schematic of transformation apparatus. (B) Schematic of resistance apparatus.

Fig. 2. Photograph of transformation equipment.

Fig. 3. Photograph of tempering equipment.

Fig. 4. (A) Schematic of sample-holder. (B) Sample dimensions.

Fig. 5. Photograph of sample-holder.

Fig. 6. Schematic of electrical circuit for resistance apparatus.

Fig. 7. Percent resistance change vs tempering time for 1090 steel at 100°C, 150°C and 180°C.

Fig. 8. Percent resistance change vs tempering time for 1046 steel at 100°C, 150°C and 180°C.

Fig. 9. Percent resistance change vs tempering time for 4340 steel at 100°C, 150°C and 180°C.

Fig. 10. Percent resistance change vs tempering time for 300-M steel at 100°C, 150°C and 180°C.

Fig. 11. Percent resistance change vs tempering time for 4340 + 3% Si at 100°C, 150°C and 180°C.

Fig. 12. Absolute resistance change vs tempering time for 1046, 4340, 300-M and 4340 + 3% Si at 100°C, 150°C and 180°C.

Fig. 13. Percent resistance change vs tempering time for 1090 steel at 240°C, 270°C and 300°C.

Fig. 14. Percent resistance change vs tempering time for 1046 steel at 240°C, 270°C and 300°C.

Fig. 15. Percent resistance change vs tempering time for 4340 steel at 240°C, 270°C and 300°C.

Fig. 16. Percent resistance change vs tempering time for 300-M steel at 240°C, 270°C and 300°C.

Fig. 17. Percent resistance change vs tempering time for 4340 + 3% Si steel at 240°C, 270°C and 300°C.

Fig. 18. Relative ductility of 1046 steel coils tempered at 180°C with time.

Fig. 19. Relative ductility of 4340 steel coils tempered at 180°C with time.

Fig. 20. Relative ductility of 300-M steel coils tempered at 180°C with time.

Fig. 21. Relative ductility of 4340 + 3% Si steel coils tempered at 180°C with time.

Fig. 22. Relative ductility of 1046, 4340, 300-M and 4340 + 3% Si steel coils tempered at 100°C for 1 hr.

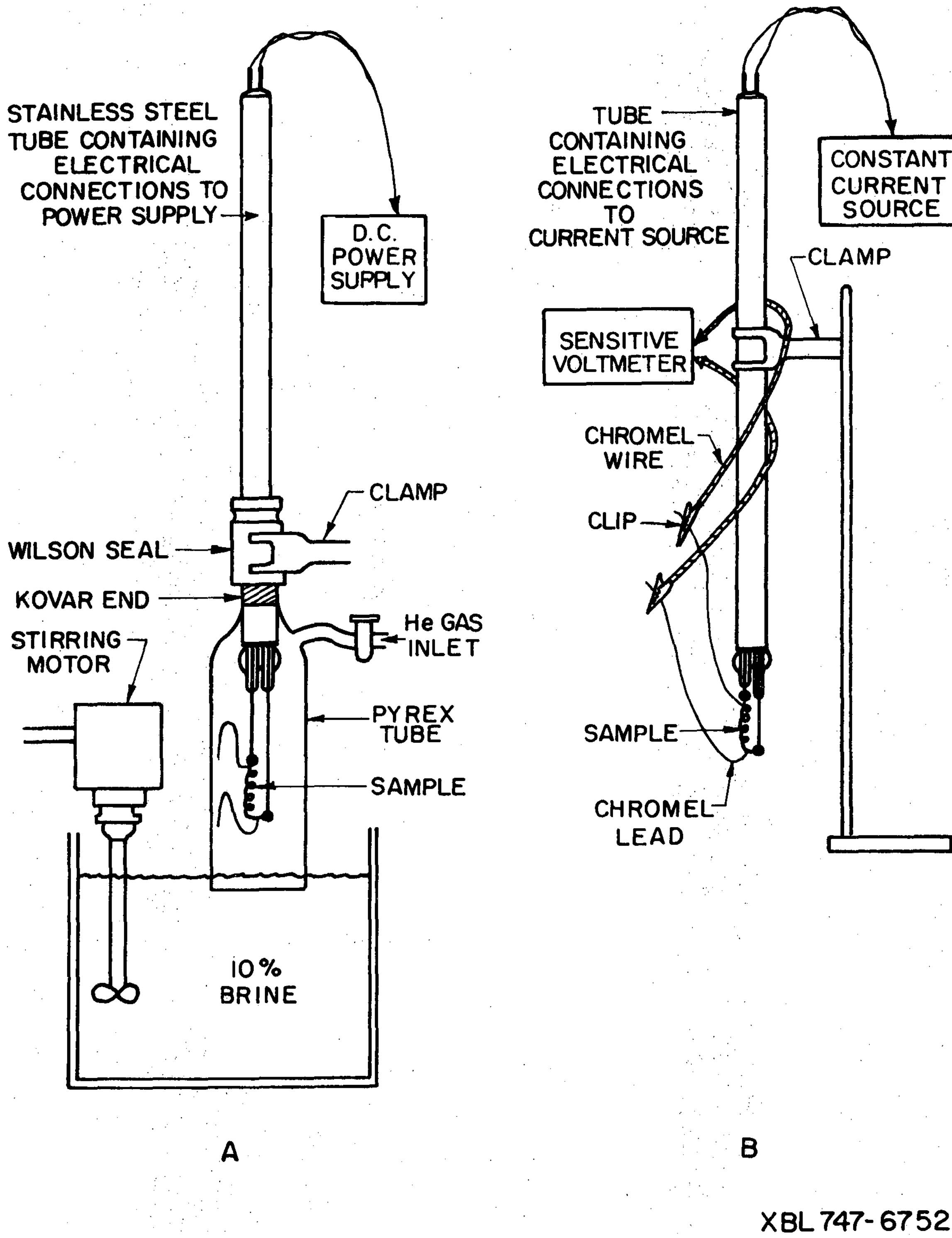
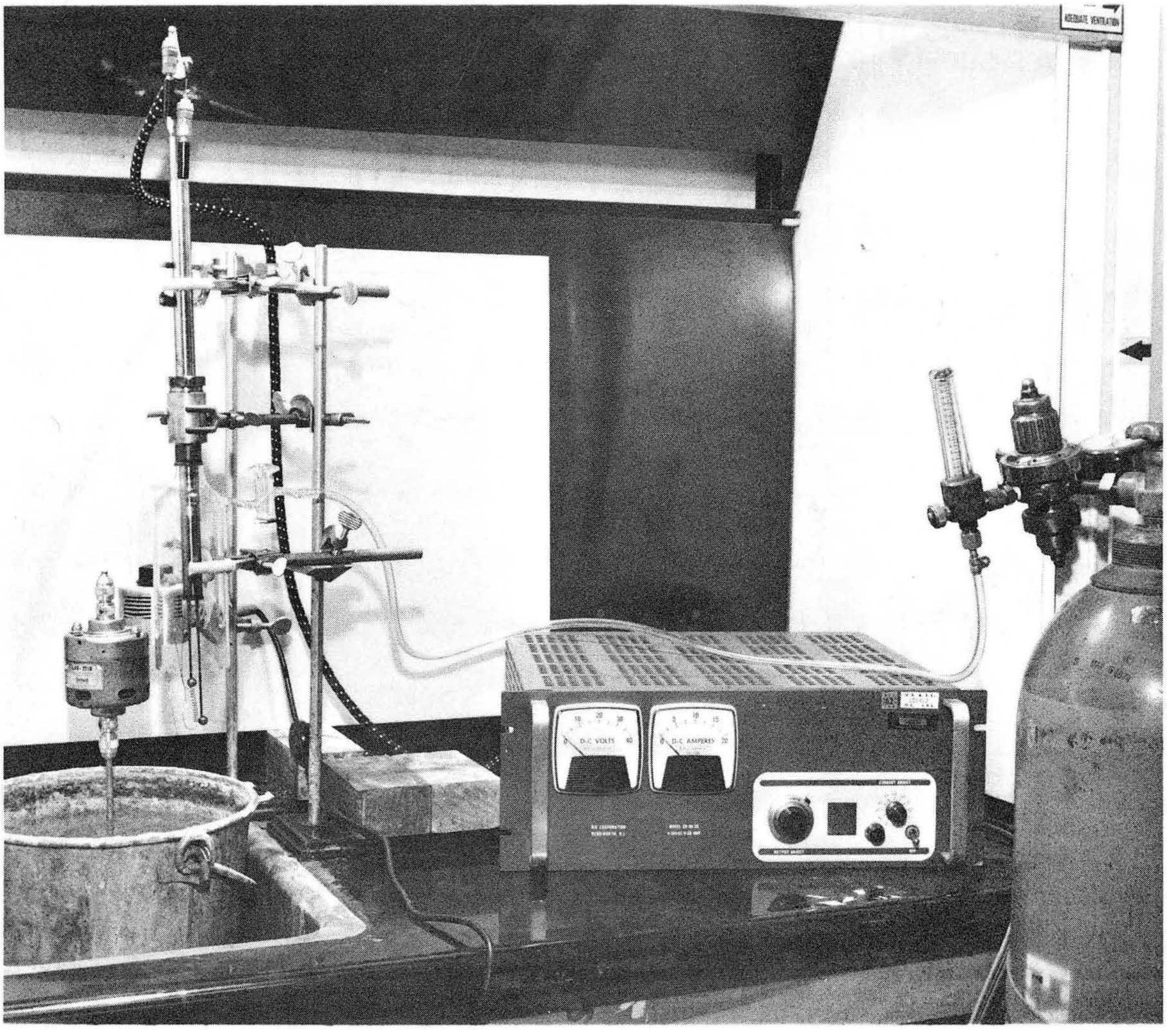
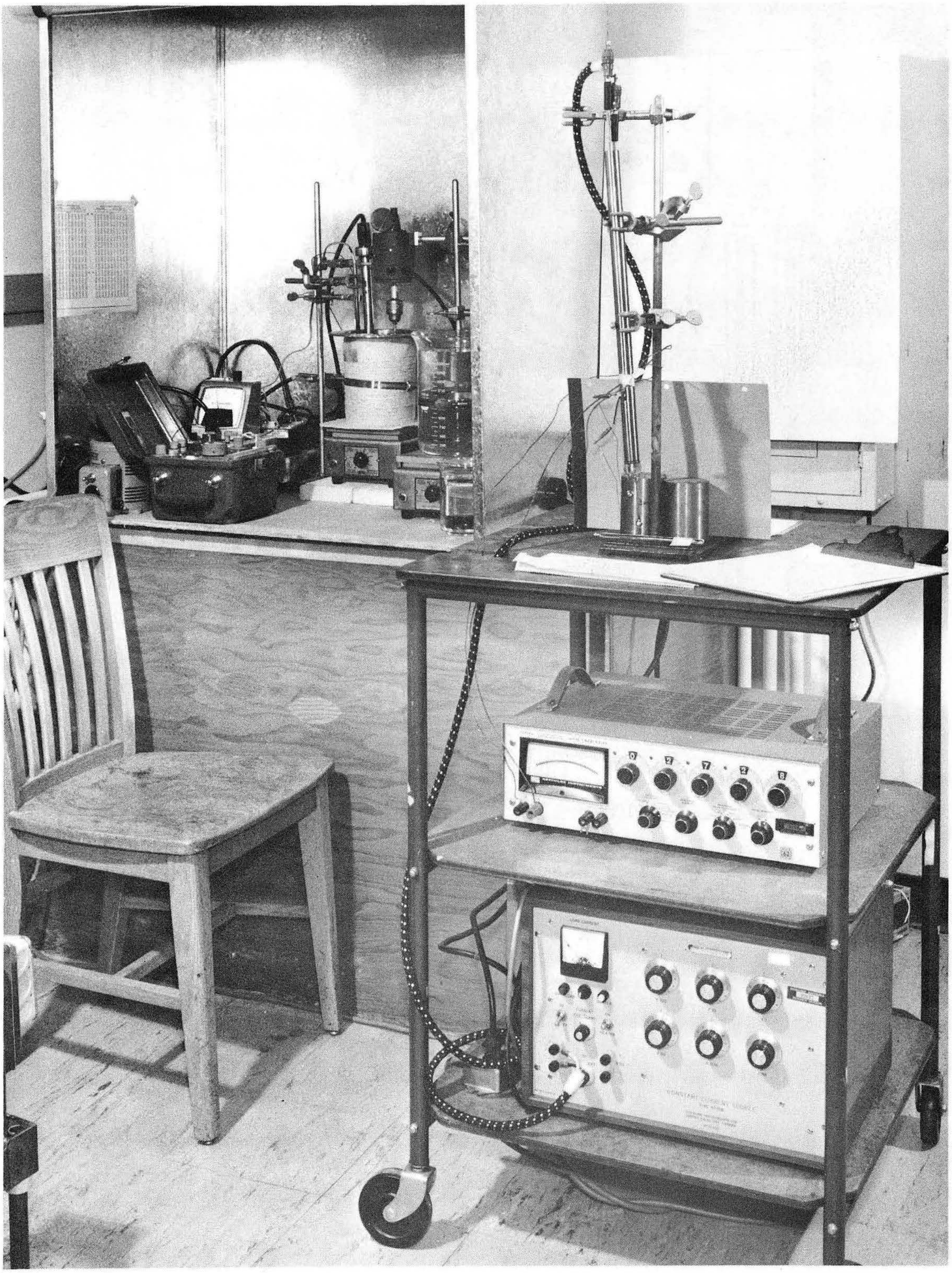

STAINLESS STEEL TUBE CONTAINING ELECTRICAL CONNECTIONS TO POWER SUPPLY
D.C. POWER SUPPLY
CLAMP
WILSON SEAL
KOVAR END
STIRRING MOTOR
He GAS INLET
PYREX TUBE
SAMPLE
10% BRINE
A
TUBE CONTAINING ELECTRICAL CONNECTIONS TO CURRENT SOURCE
CONSTANT CURRENT SOURCE
CLAMP
SENSITIVE VOLTMETER
CHROMEL WIRE
CLIP
SAMPLE
CHROMEL LEAD
B
XBL 747-6752

Fig. 1.



XBB 747-4613

Fig. 2.



XBB 747-4611

Fig. 3.

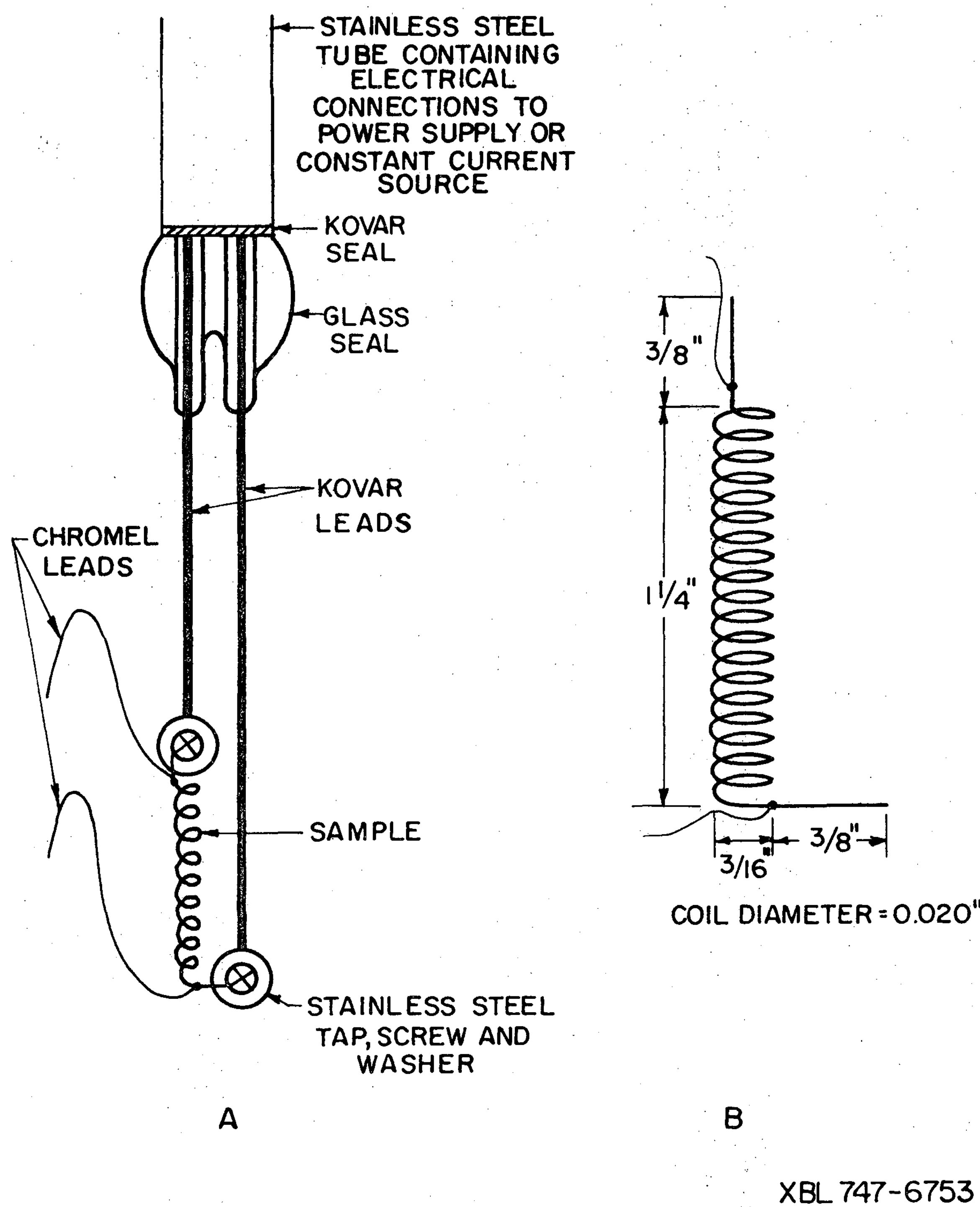
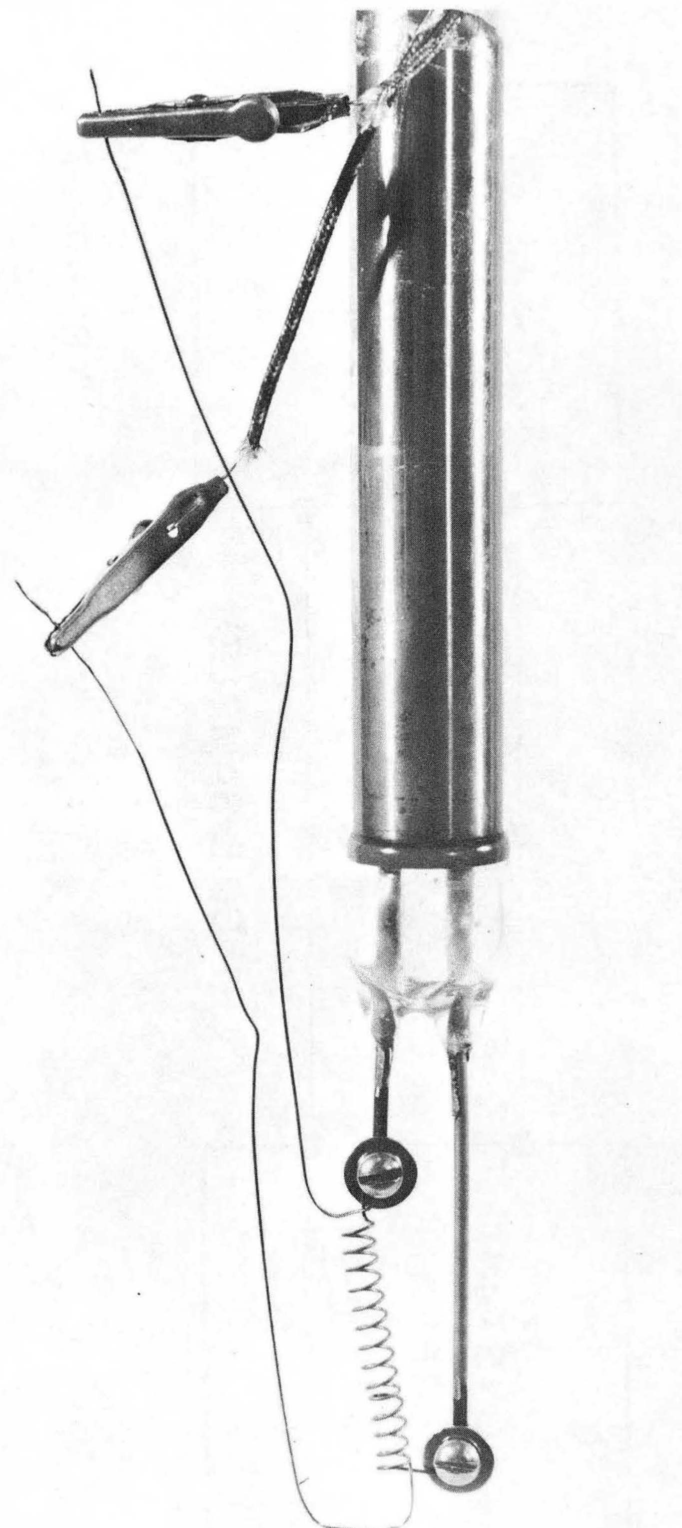
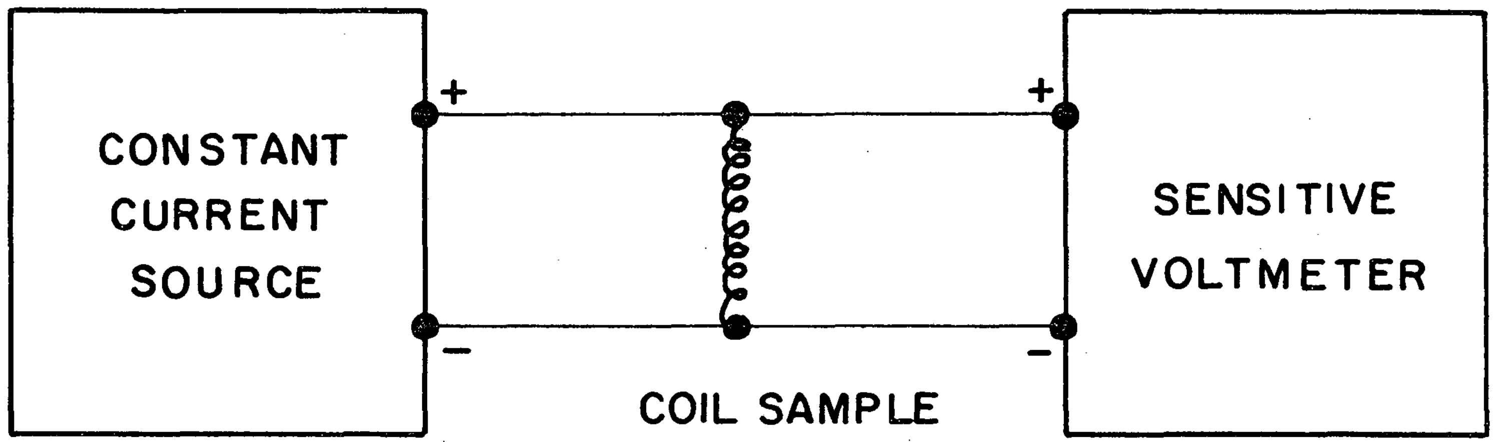

STAINLESS STEEL TUBE CONTAINING ELECTRICAL CONNECTIONS TO POWER SUPPLY OR CONSTANT CURRENT SOURCE
KOVAR SEAL
GLASS SEAL
KOVAR LEADS
CHROMEL LEADS
SAMPLE
STAINLESS STEEL TAP, SCREW AND WASHER
A
3/8"
1 1/4"
3/16"
3/8"
COIL DIAMETER = 0.020"
B
XBL 747-6753

Fig. 4.



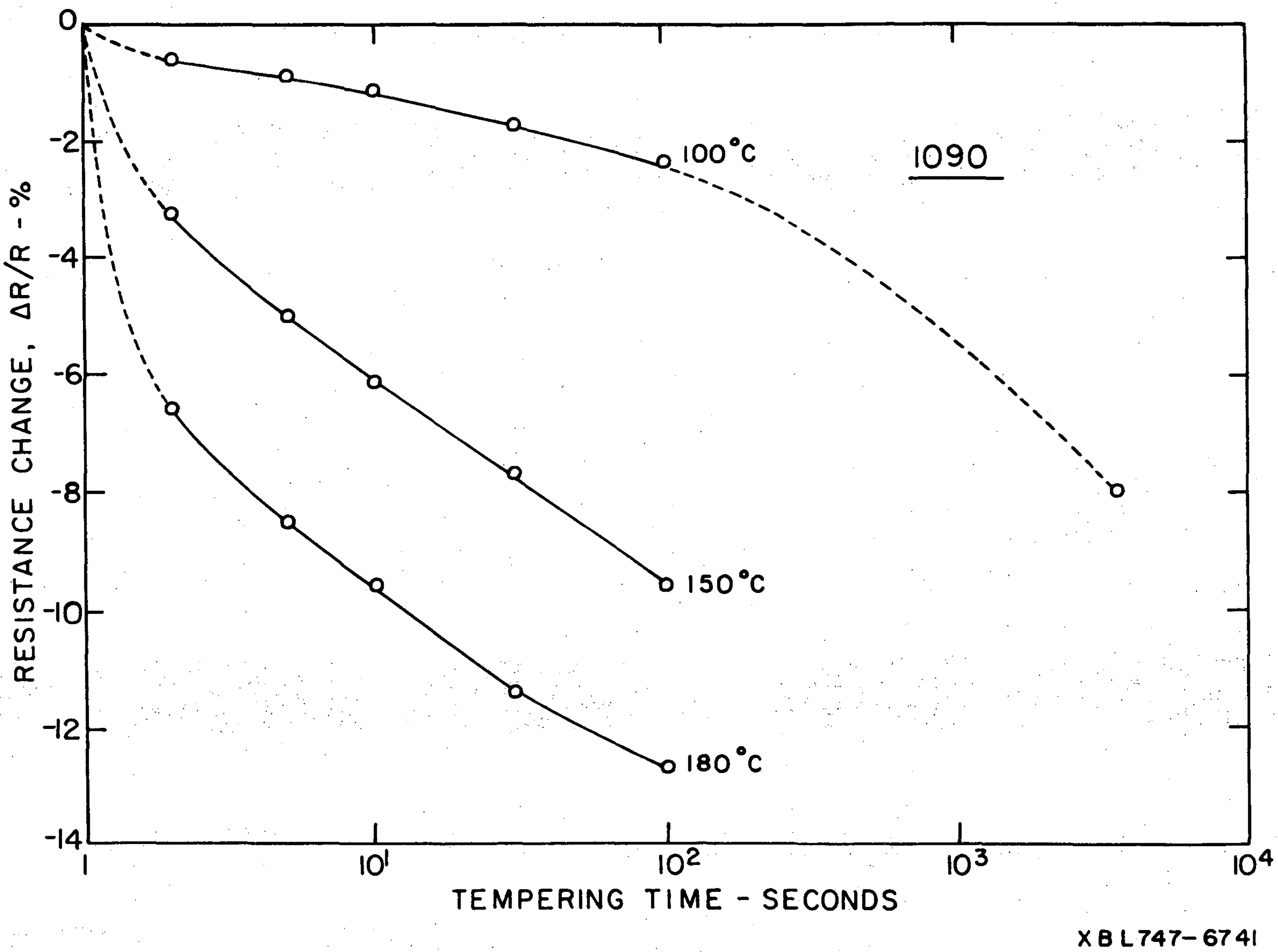
XBB 747-4612

Fig. 5.



XBL747-6754

Fig. 6.



1090
100°C
150°C
180°C
0
-2
-4
-6
-8
-10
-12
-14
RESISTANCE CHANGE, ΔR/R - %
1
10¹
10²
10³
10⁴
TEMPERING TIME - SECONDS
XBL747-6741

Fig. 7.

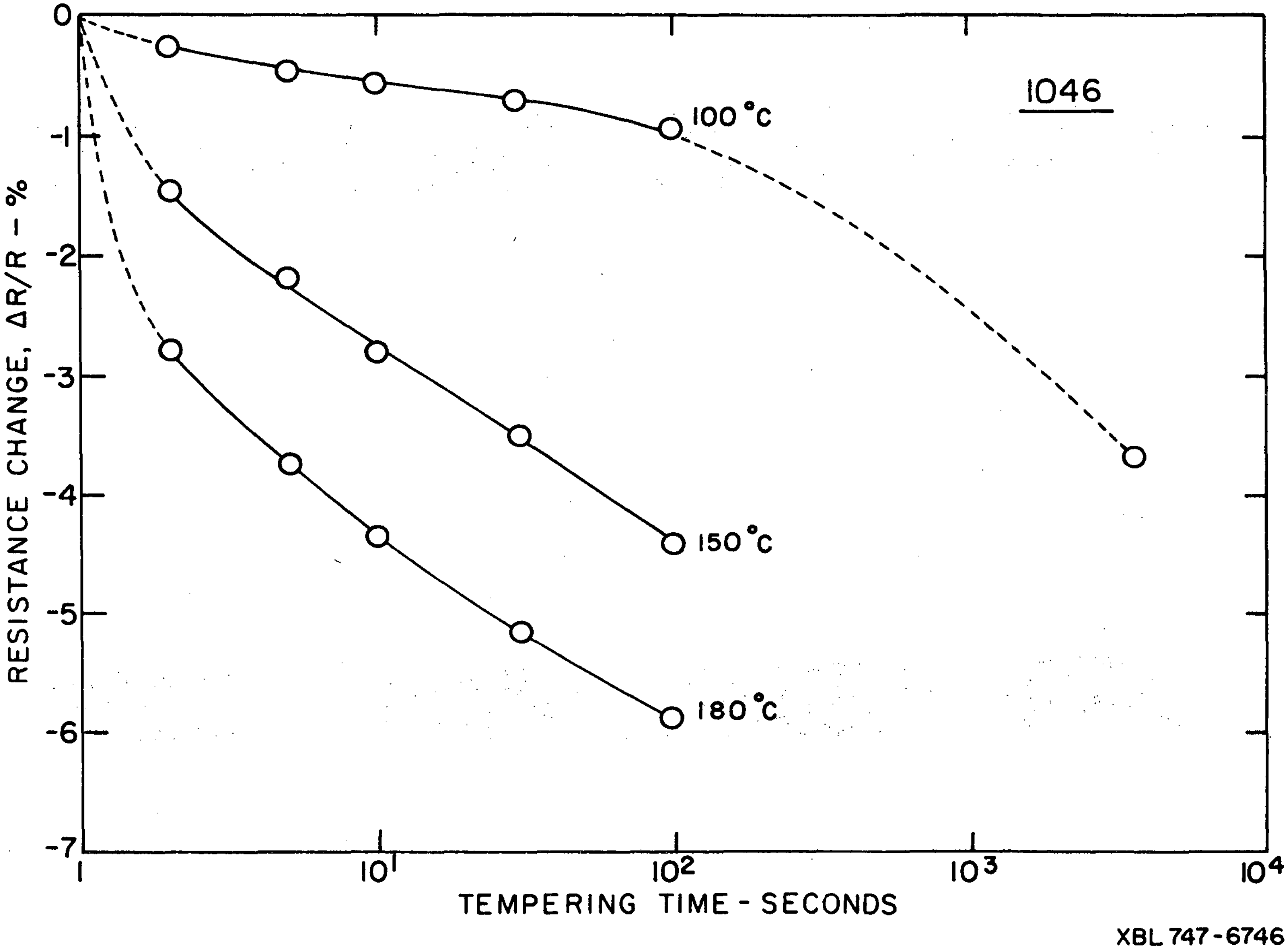
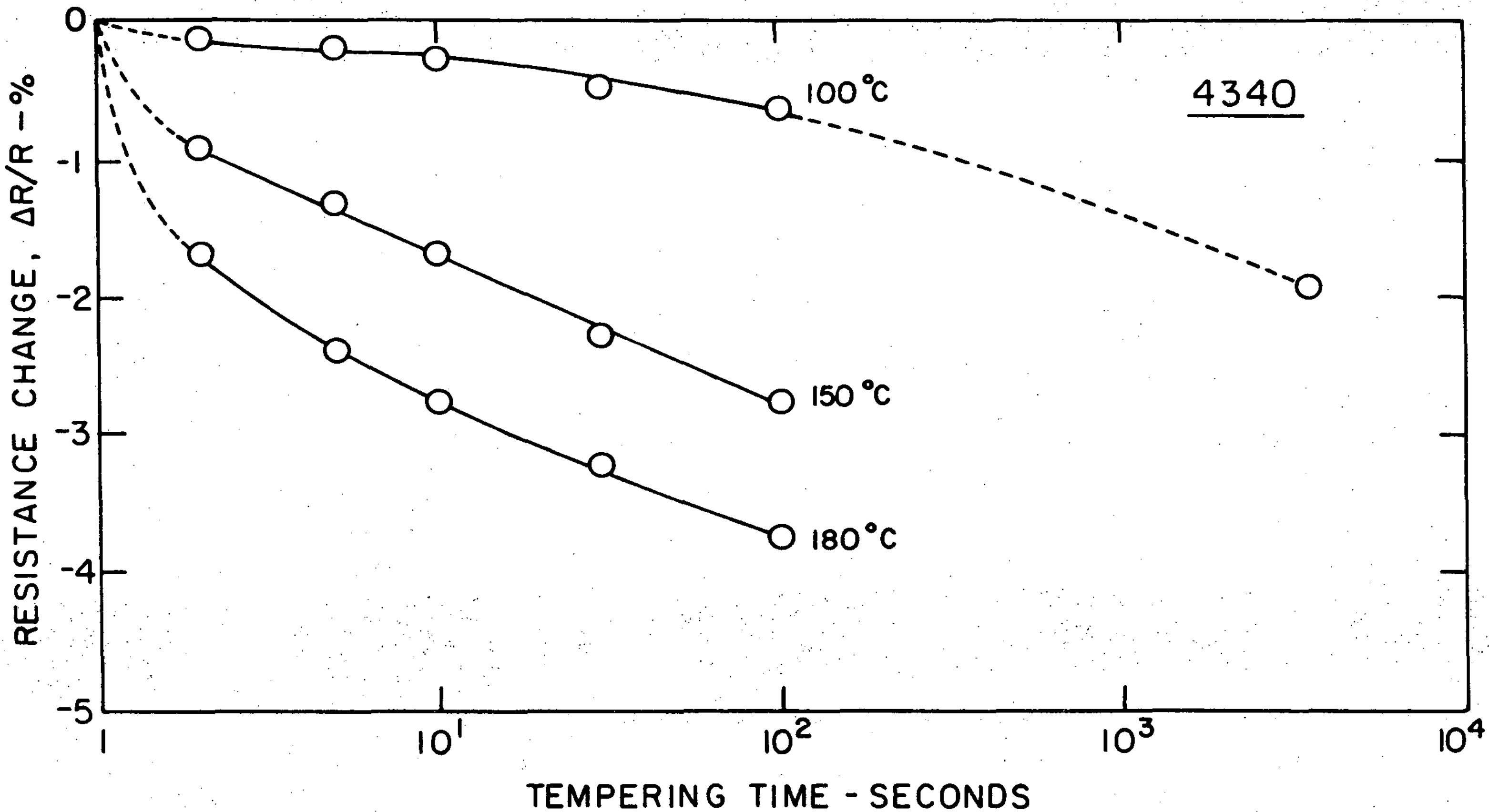
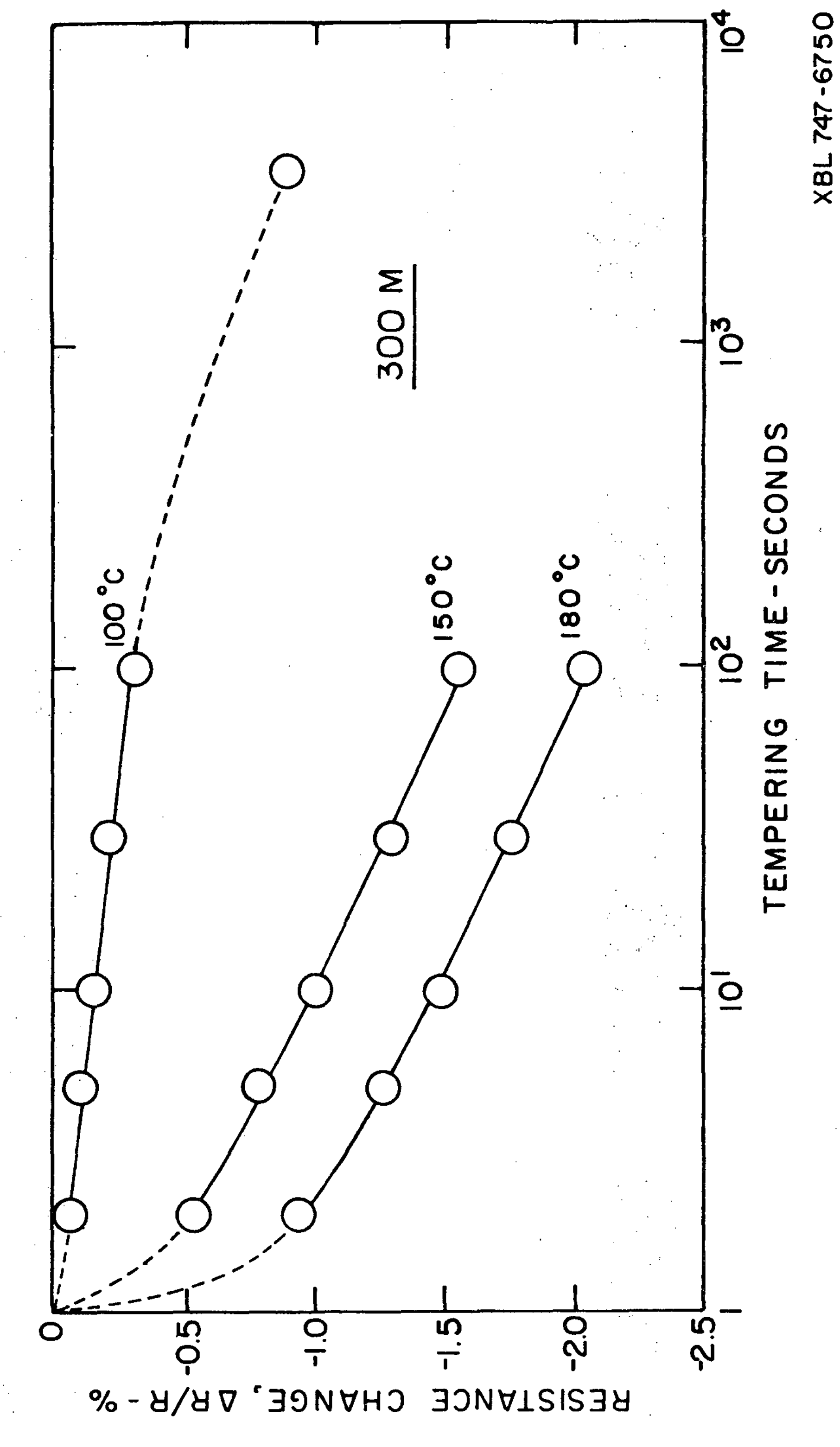

1046
100 °C
150 °C
180 °C
RESISTANCE CHANGE, ΔR/R – %
0
-1
-2
-3
-4
-5
-6
-7
TEMPERING TIME - SECONDS
1
10¹
10²
10³
10⁴
XBL 747-6746

Fig. 8.



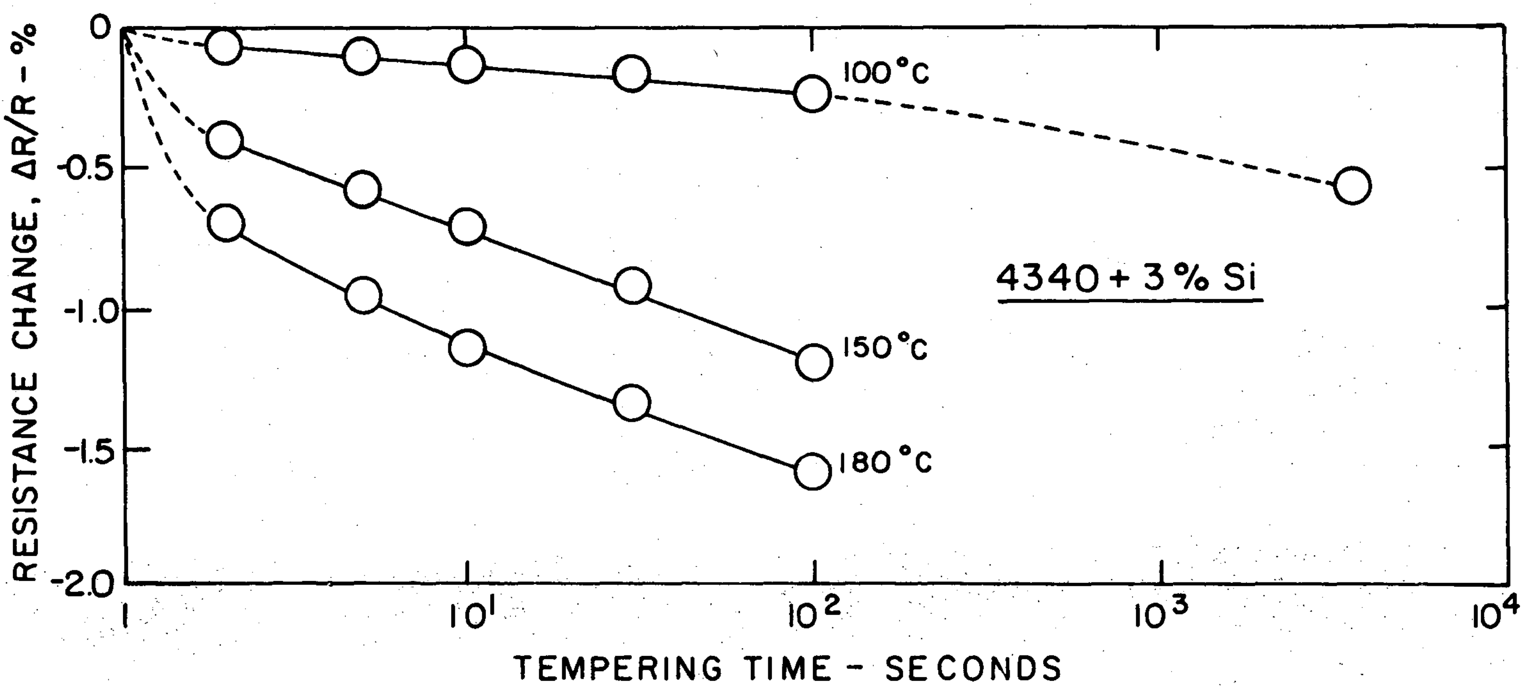
XBL 747-6744

Fig. 9.



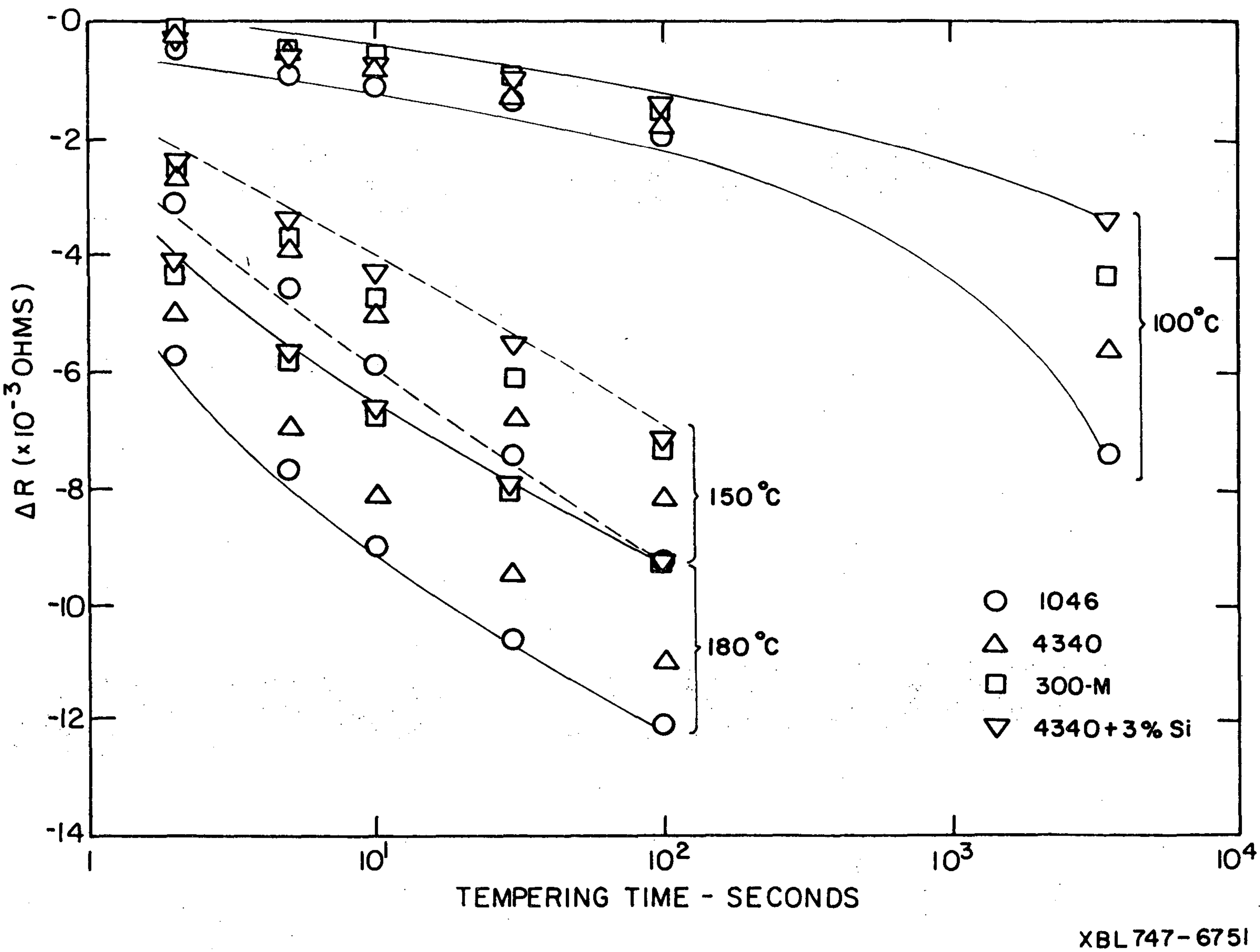
RESISTANCE CHANGE, ΔR/R - %
0
-0.5
-1.0
-1.5
-2.0
-2.5
TEMPERING TIME - SECONDS
1
10^1
10^2
10^3
10^4
100°C
150°C
180°C
300 M
XBL 747-6750

Fig. 10.



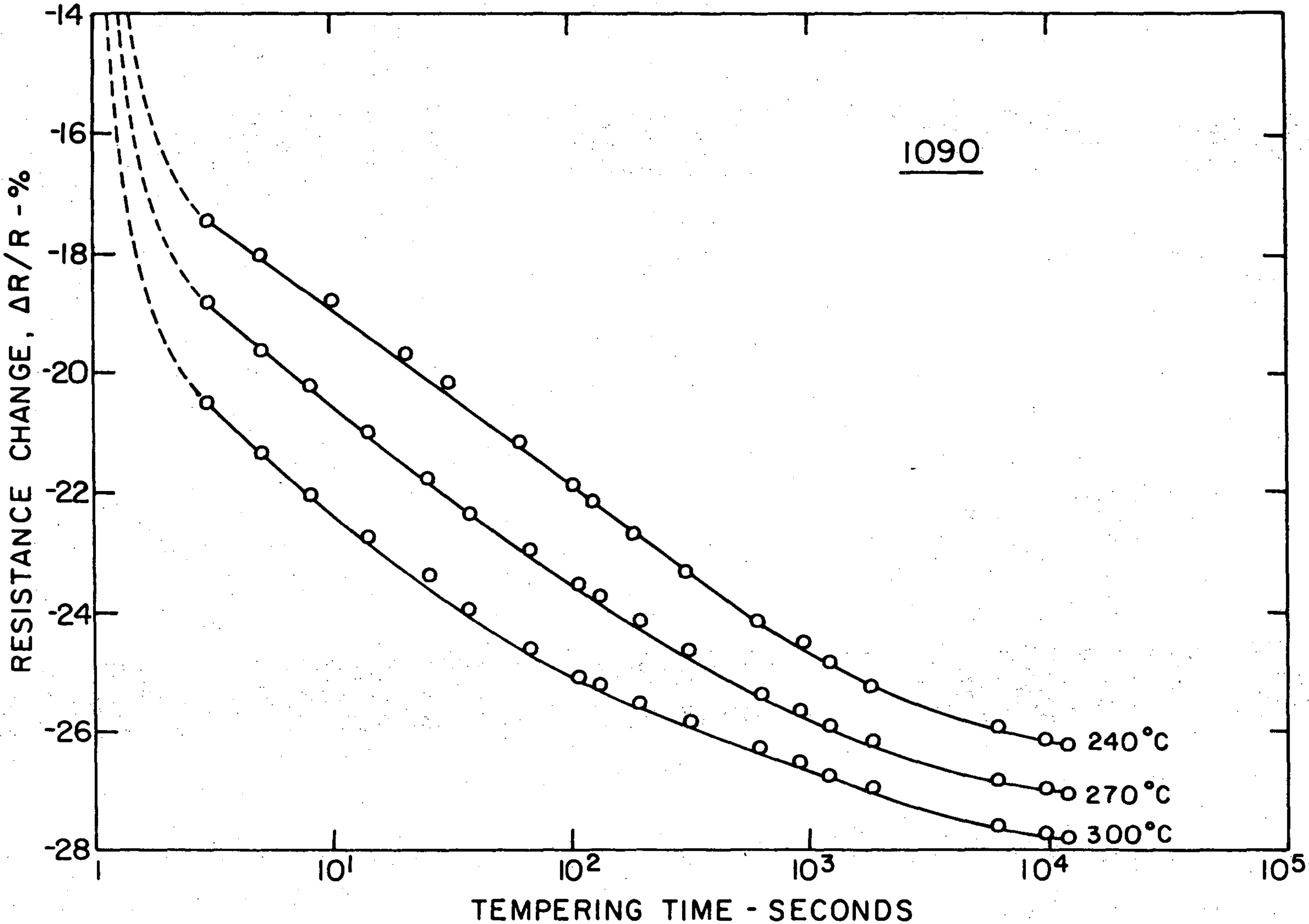
XBL747-6748

Fig. 11.



-0
-2
-4
-6
-8
-10
-12
-14
ΔR (x10^-3 OHMS)
1
10^1
10^2
10^3
10^4
TEMPERING TIME - SECONDS
100°C
150°C
180°C
1046
4340
300-M
4340+3% Si
XBL 747-6751

Fig. 12.



XBL 747-6742

Fig. 13.

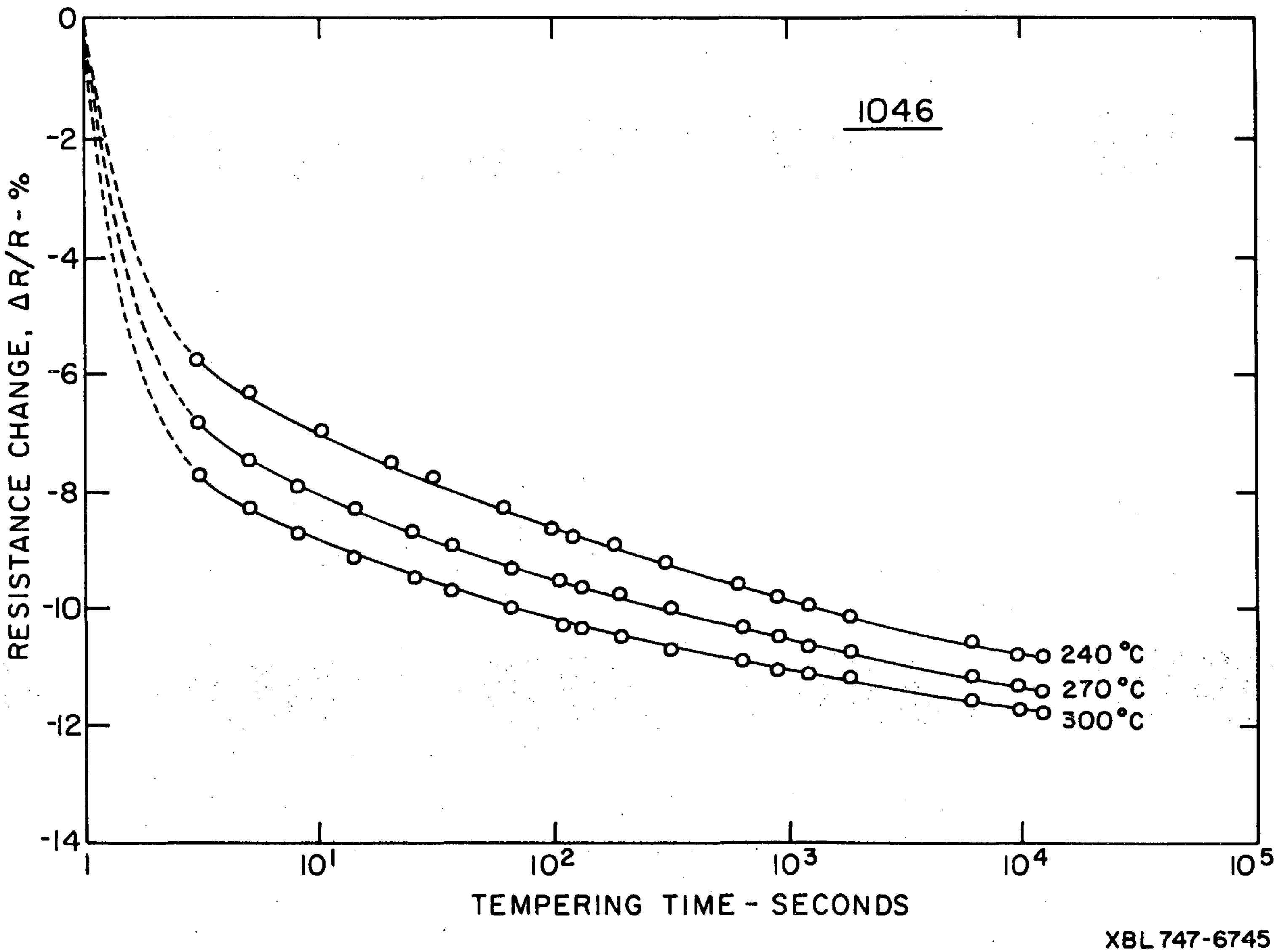
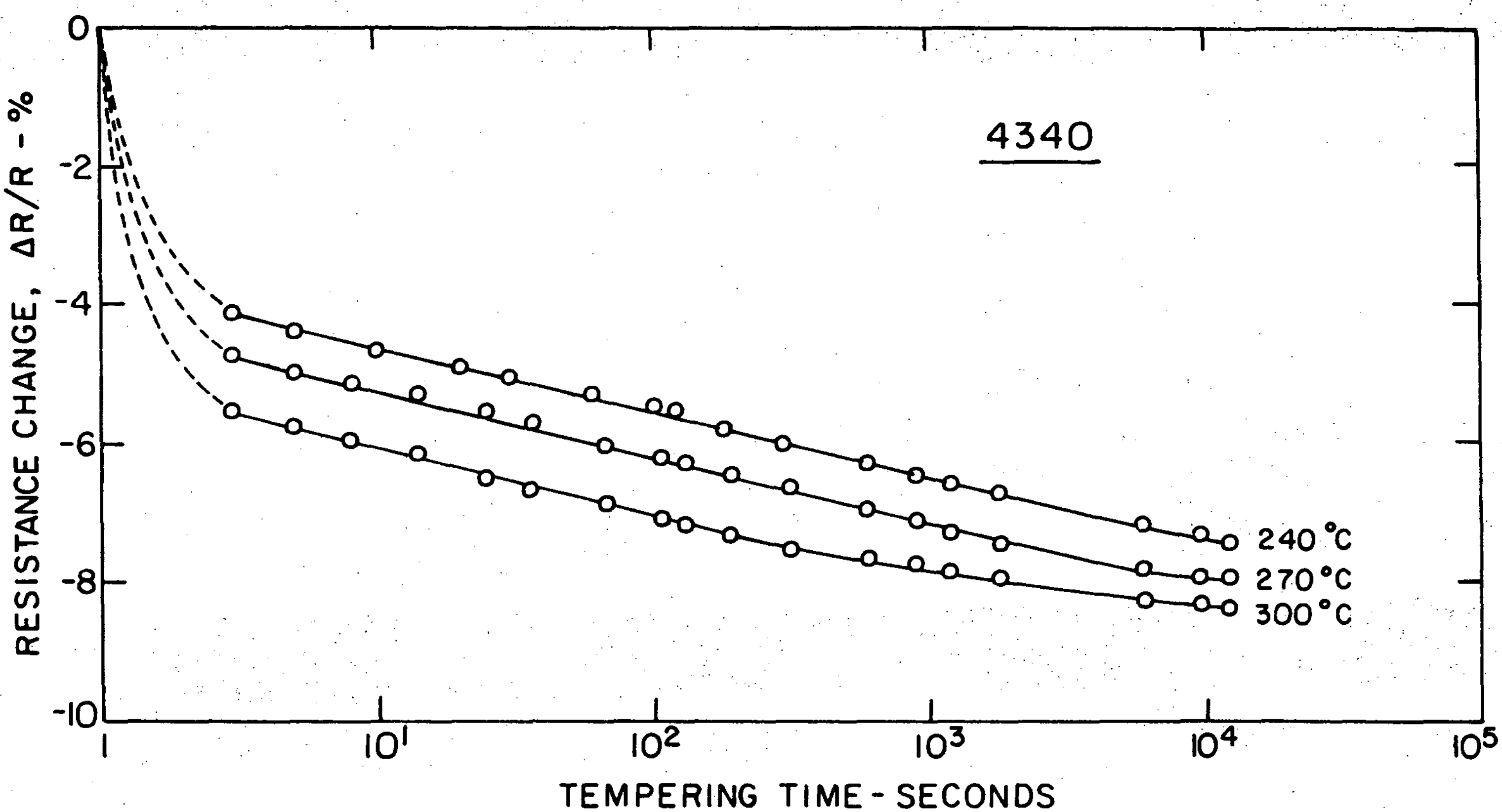

1046
RESISTANCE CHANGE, ΔR/R - %
0
-2
-4
-6
-8
-10
-12
-14
1
10^1
10^2
10^3
10^4
10^5
TEMPERING TIME - SECONDS
240 °C
270 °C
300 °C
XBL 747-6745

Fig. 14.



XBL 747-6743

Fig. 15.

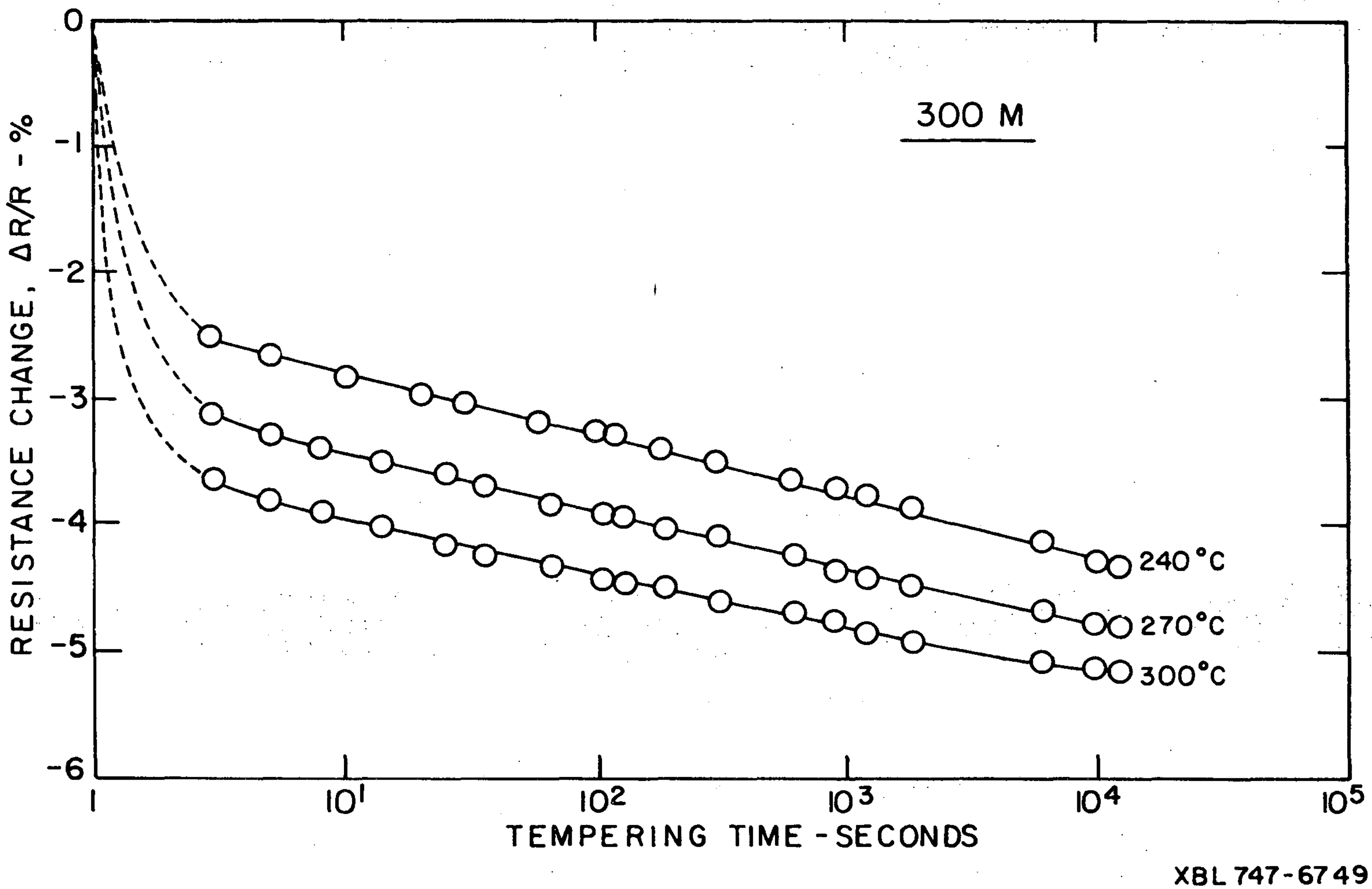
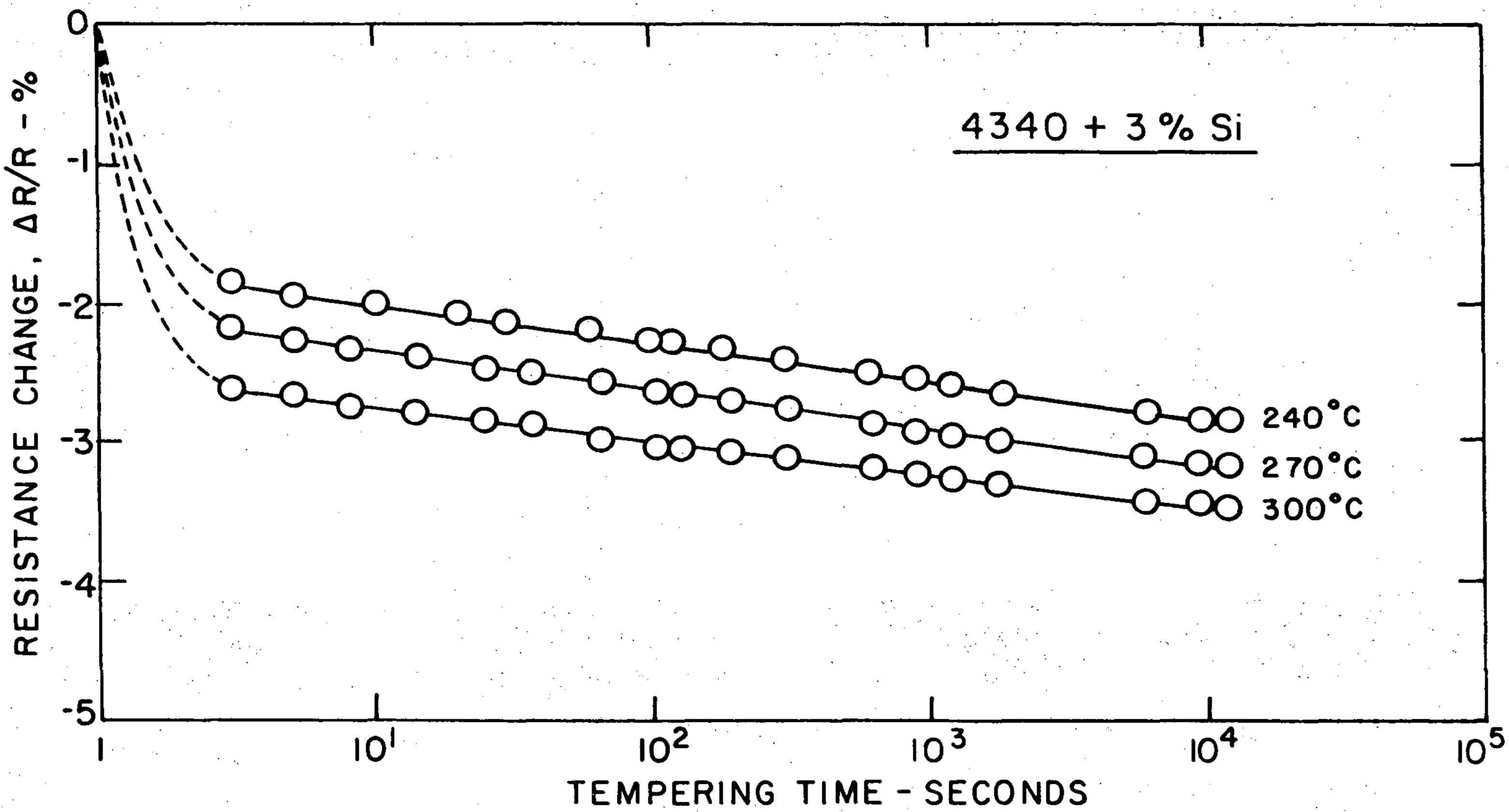
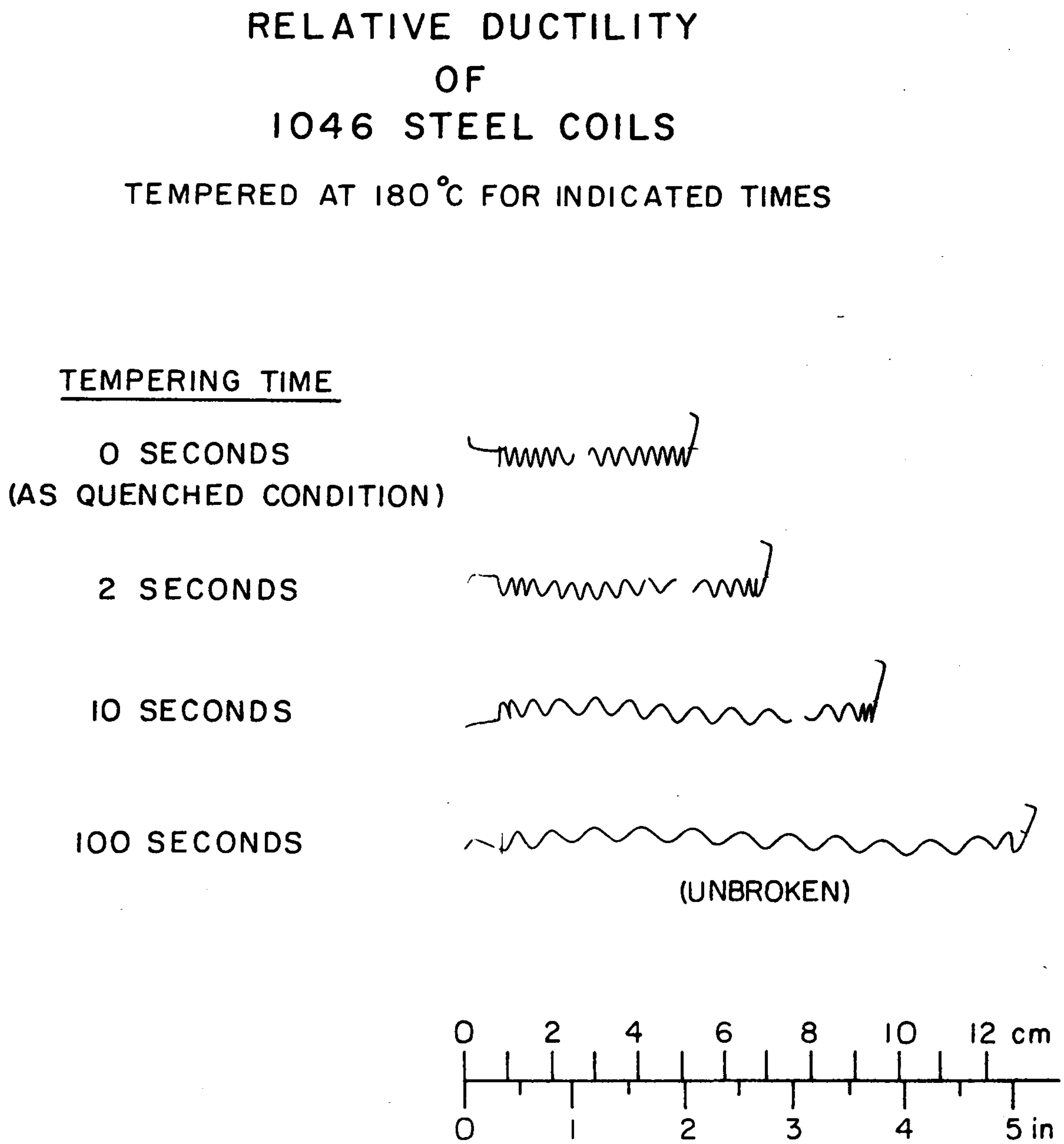

300 M
RESISTANCE CHANGE, ΔR/R - %
0
-1
-2
-3
-4
-5
-6
1
10¹
10²
10³
10⁴
10⁵
TEMPERING TIME - SECONDS
240°C
270°C
300°C
XBL 747-6749

Fig. 16.



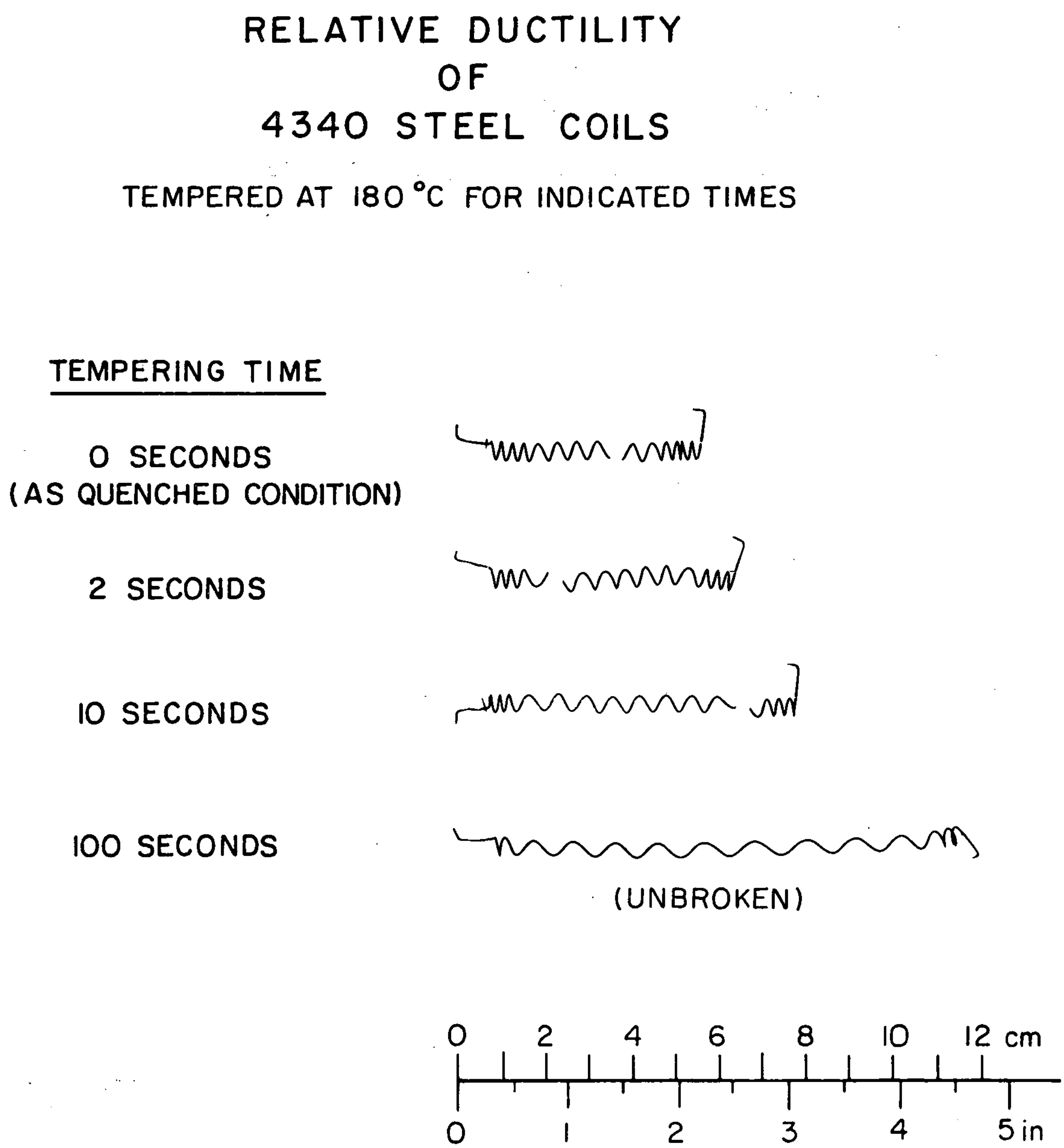
XBL 747-6747

Fig. 17.



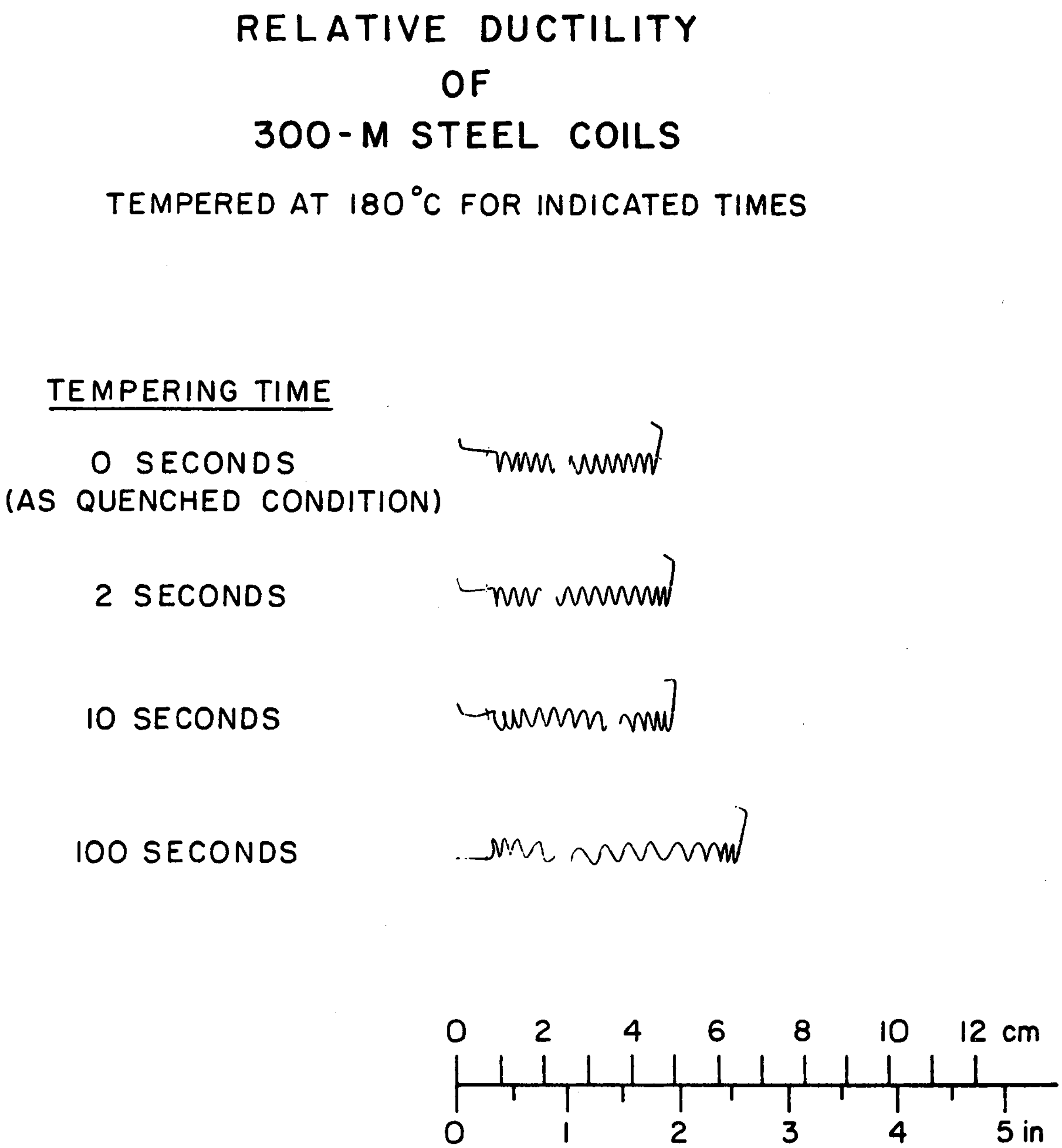
XBB 747-4581

Fig. 18.



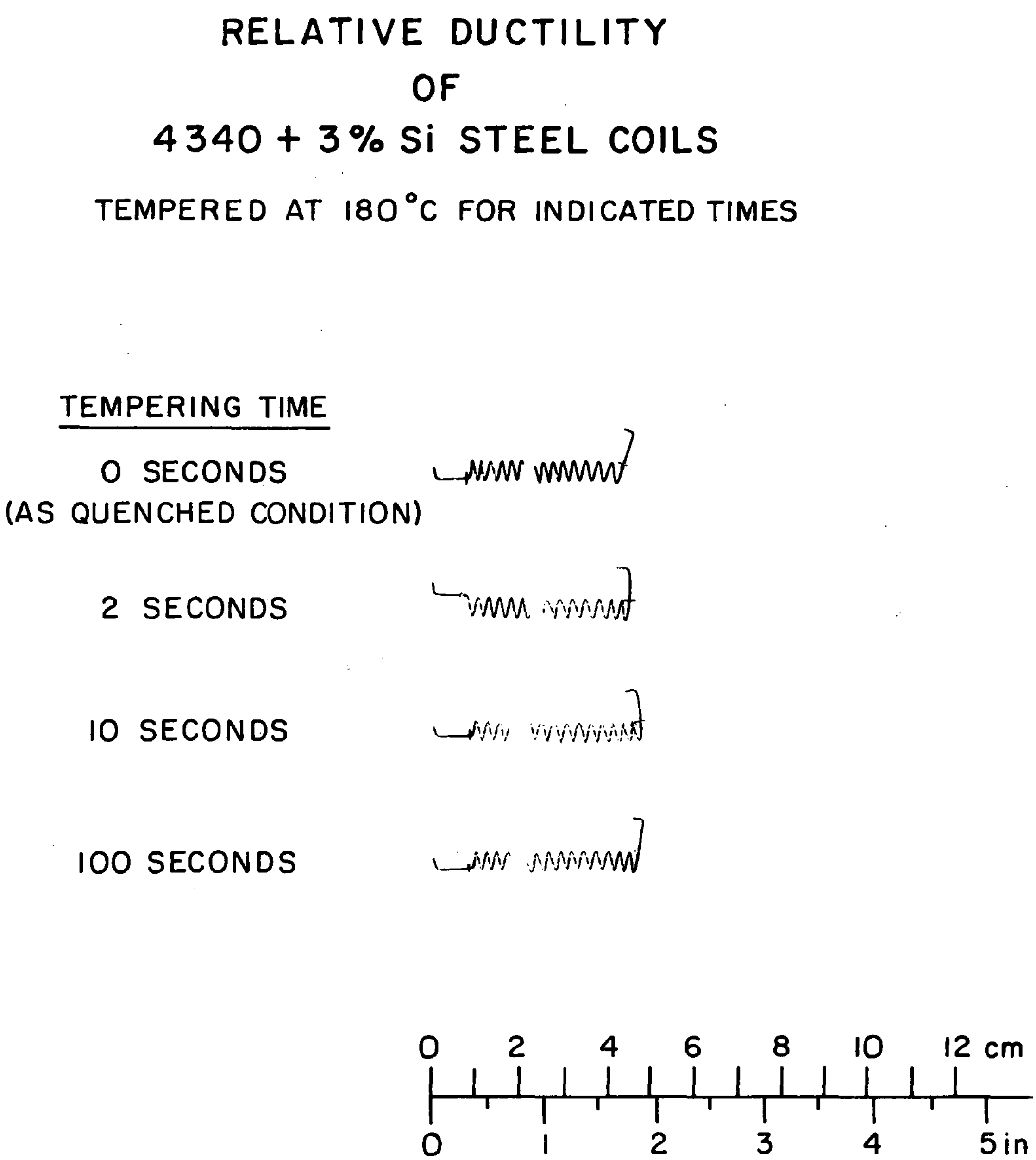
XBB 747-4585

Fig. 19.



XBB 747-4582

Fig. 20.

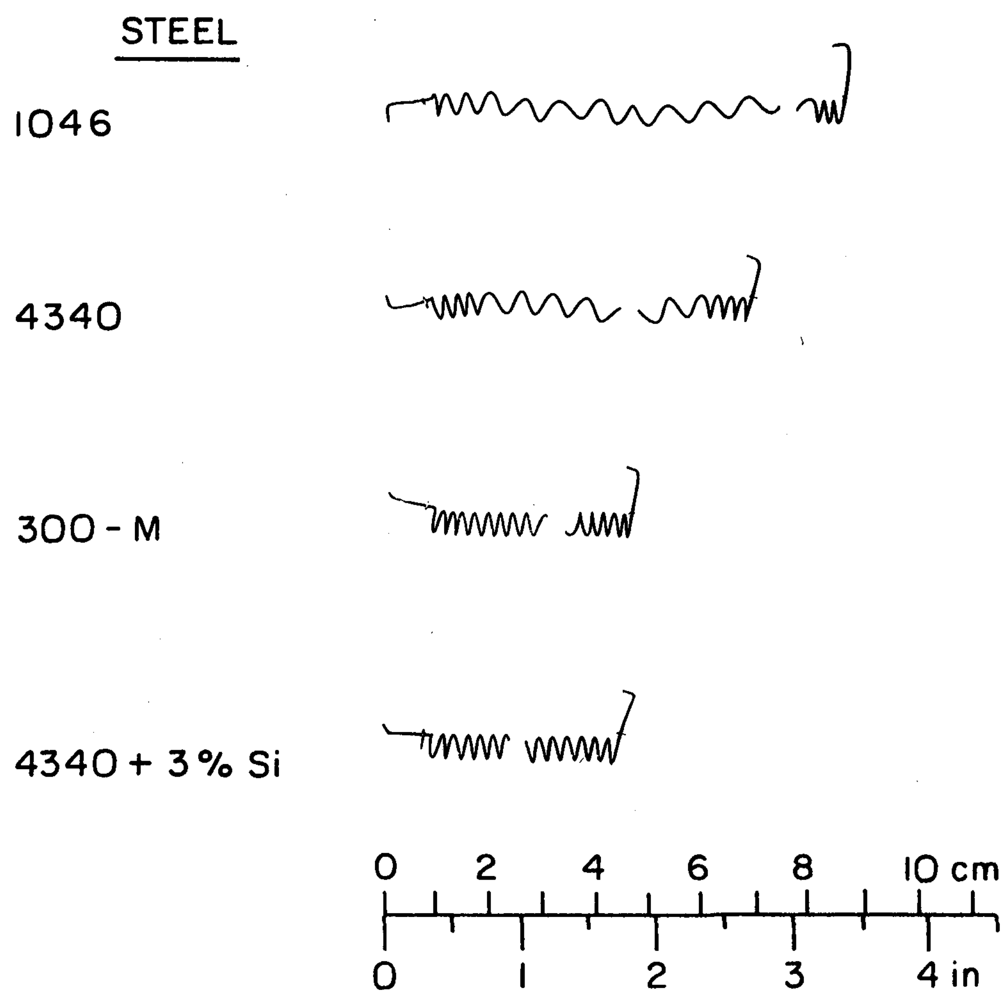


XBB 747-4583

Fig. 21.

RELATIVE DUCTILITY OF STEEL COILS

TEMPERED AT 100 °C FOR ONE HOUR



XBB 747-4584

Fig. 22.