

# EFFECT OF PRIOR COLD WORK ON THE HIGH-TEMPERATURE PROPERTIES OF A CHROMIUM-MOLYBDENUM STEEL

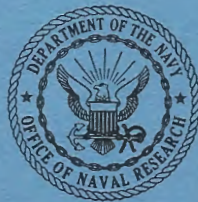
Paul Shahinian

High Temperature Alloys Branch  
Metallurgy Division

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ABSTRACT

The effect of cold work on the microstructure and mechanical properties of a polycrystalline metal was investigated by means of metallographic and mechanical tests. The material was cold worked by rolling at 70% reduction at 700° F, 800° F, and 900° F. The microstructure was examined by metallographic techniques and the mechanical properties were determined by tensile and impact tests. The results show that cold work increases the strength and hardness of the metal and decreases its ductility and impact toughness. The microstructure becomes more refined and dislocation density increases with increasing cold work.

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AUTHORIZATION

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## ABSTRACT

The effect of cold work on the high-temperature properties of a quenched and tempered chromium-molybdenum steel was investigated by means of stress-rupture and relaxation tests. The material with several levels of cold reduction (0, 8, 15 and 39% of cross-sectional area) was tested in stress-rupture at 700°, 800°, 900°, and 1000° F (370°, 425°, 480°, and 540° C) in the stress range of 45,000 to 128,000 psi, and in relaxation at 900° F (480° C) and 80,000 psi initial stress.

The rupture and creep strengths of the steel at 700° F (370° C) was greatly enhanced by cold work, the improvement increasing with larger amounts of cold work. At 800° and 900° F (425° and 480° C) the rupture and creep strengths were generally lowered by critical amounts of cold deformation (8 to 15 %), while beyond the critical values increased strengths were obtained. An explanation for this behavior is given in terms of the Bauschinger effect. There was no apparent influence of prior deformation on rupture strength at 1000° F (540° C); however, creep strength decreased generally with increasing prior deformation. In relaxation at 900° F (480° C) residual stress was lowered by an increase in cold work.

## PROBLEM STATUS

This is a final report on the effect of cold deformation on strength of steel at elevated temperatures; no further work is expected to be done on this phase of the problem.

## AUTHORIZATION

NRL Problem No. M01-09

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## EFFECT OF PRIOR COLD WORK ON THE HIGH-TEMPERATURE PROPERTIES OF A CHROMIUM-MOLYBDENUM STEEL

### INTRODUCTION

A knowledge of the effect of metallurgical variables on the high-temperature properties of metals is essential for the proper design of parts for service above room temperature. There is a particular need for information on the influence of cold deformation on the variation of strength with time of steel at elevated temperatures for the design of aircraft engine bolts. For example, the threads on some commercial bolts are formed by cold-rolling, and it is not known whether this cold work is beneficial or deleterious. In addition to being of practical importance, the behavior of metals with prior cold work is of fundamental interest.

A number of investigations have been made, particularly on non-ferrous metals and alloys (1-8), of the effect of cold work on creep resistance at high temperatures. The results of these studies agree, in general, that prior cold work increases creep strength; however, the benefit from cold work is lost at temperatures where rapid recovery or recrystallization occur. In the case of Monel, there was found to be an optimum degree of cold work for improving the strength at each temperature; the optimum percentage of cold work was noted to decrease with increasing temperature (8). A similar effect has been observed with stainless steel (9). The improved creep resistance due to cold work of a high-temperature alloy, low-carbon N-155, was attributed (10) to internal stresses in the crystal lattice. Very little information is available on the effect of prior deformation on the creep properties of carbon and low-alloy steels. In a study (11) on a 0.40% carbon steel in the hot-rolled condition at 600° - 1000° F (315° - 540° C), previous cold work (6 and 12 % elongation) was found to lower creep resistance. Although the latter results are not directly comparable with data on other materials, there is doubt as to the effect of cold work on the behavior of steel at elevated temperatures.

It was the object of this investigation to determine the influence of prior cold-deformation on the high-temperature strength of a chromium-molybdenum steel. Creep and rupture properties were

evaluated by stress-rupture tests and, to a limited extent, the variation of strength with time was determined by stress-relaxation tests.

## EXPERIMENTAL PROCEDURE

The investigation was conducted with a single bar of commercial steel of the following chemical composition: 0.38% C, 0.45% Mn, 1.57% Cr, 0.48% Mo, 0.70% Si, 0.008% P, 0.015% S. The material was received as hot-rolled 5/8-inch-diameter rods and was heat-treated prior to cold-working. The treatment consisted of heating at 1800° F (980° C) for 1 hour, oil quenching, and tempering at 1200° F (650° C) for 2 hours. Several levels of cold reduction, 8, 15, and 39 % of cross-sectional area, were obtained by means of rolling. Bar sections of approximately 15 inches were rolled on six sides for all reductions except for the case of the 39 % reduction samples, which were rolled on four sides. Reduction-of-area measurements were made on each specimen; the variation from the values indicated was no more than  $\pm 1$  %.

Room-temperature tensile tests were made on the material with various degrees of cold work as well as without cold work. Standard 0.357-inch-diameter specimens were used except for the 39 % cold-worked material which permitted a maximum specimen diameter of 0.250 inch.

Stress-rupture tests were conducted on the steel with 0, 8, 15, and 39 % cold reduction at 700°, 800°, 900°, and 1000° F (370°, 425°, 480°, and 540° C) in the stress range of 45,000 to 128,000 psi. In addition, in order to determine if tempering would alter the influence of cold work on rupture properties, specimens with 8, 15 and 39 % cold reduction were tempered for 3 hours at 1200° F and then tested in stress-rupture at 800° F and 100,000 psi. The specimens employed had a 0.250-inch diameter and a 1.25-inch gage length, and were machined from the centers of the bars. Conventional single-lever, constant-load, stress-rupture machines were used. Temperatures were measured by a chromel-alumel thermocouple placed in contact with the center position of the specimen length, and the temperatures were maintained to within  $\pm 2$ ° F. The temperature gradient did not exceed 2° F along the gage length of the specimen. The specimens were held at temperature for 1 hour prior to loading. Extension of each specimen was automatically recorded from the deflection of the loading lever, giving an elongation-versus-time curve; in addition, periodic readings were taken with a dial gage. Single tests were run, generally, for each condition of temperature, stress, and degree of cold work.

A series of specimens (stress-rupture type) with various degrees of cold work was tested in stress relaxation at 900° F (480° C). An initial stress of 80,000 psi was applied and the relaxation in stress

recorded for 500 hours. The tests were conducted in a machine normally used for constant strain rate tests. In operation, the tensile load is applied rapidly by means of a motor-driven cam to the desired load level; the over-all strain is then held constant. A load cell placed in the grip system of the specimen permits the recording of the variation of load with time. The amount of elastic strain experienced by the loading members is small because of their large size. The strain in the specimen is constant except for possibly a minor change due to elastic recovery of the massive loading members. Comparison of relaxation curves obtained from this machine with those from a conventional relaxation machine showed no appreciable difference (12). After completion of the relaxation tests (500 hours), the specimens were tested in stress-rupture at 900° F (480° C) and 80,000 psi.

Metallographic examinations and hardness measurements (discussed later) were made on the threaded and gage sections of some fractured specimens and on the material prior to testing. Vickers pyramid hardness measurements were obtained using a 5-kilogram load.

## RESULTS AND DISCUSSION

The room-temperature tensile properties of the quenched and tempered steel with various degrees of cold reduction are listed in Table 1. A reduction in tensile strength and in 0.2 % yield strength was observed with the 8 % cold-worked material. At the higher levels of cold work (15 and 39 %), increases in tensile and yield strengths were obtained. Elongation and reduction of area values generally decreased with increasing cold work.

TABLE 1  
Room Temperature Tensile Properties

Cold Work	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%)	Reduction of Area (%)
0%	152,000	133,600	20	62
8%	149,000	127,000	19	60
15%	154,500	135,000	19	58
39%	166,000	153,000	18	54

The effect of prior cold deformation on high-temperature properties was evaluated in terms of rupture strength, creep strength and residual stress in the case of the relaxation tests. The stress to

produce a minimum creep rate of 0.5 in./in./1000 hours was used as a measure of creep strength. Time to rupture, minimum creep rate, total elongation, and reduction of area data, listed in Table 2, were obtained from the stress-rupture tests.

TABLE 2  
Stress-Rupture Results

A - 0% Cold worked      C - 15% Cold worked  
B - 8% Cold worked      D - 39% Cold worked

Temp. (°F)	Stress (psi)	Code	Rupture Time (hr)	Min. Creep Rate (in./in./1000 hr)	Elongation (%)	Reduction of Area (%)	
700	128,000	DD03	79.0	.573	17	52	
		125,000	A2	1.5	27.2	20	64
			B2	8.2	4.77	17	61
			C2	42.9	1.0	18	64
			D2	2756.	.0266	19	52
	120,000	A02	23.1	1.76	18	59	
		B02	80.4	.636	21	60	
		C02	112.3	.49	18	59	
	800	105,000	A05	9.5	5.03	23	72
			B05	6.4	8.05	21	70
			C05	12.3	4.95	23	68
			D05	166.	.456	22	60
100,000			A5	241.1	.197	19	67
		B5	34.6	1.72	22	68	
		C5	115.7	.57	21	68	
		D5	649.3	.130	22	58	
95,000		A7	2906.	.0262	19	56	
		B7	608.6	.0795	19	64	
		C7	406.7	.176	25	67	
		D7	4270.	.0228	23	58	
900		85,000	A8	39.3	1.23	26	75
			B81	26.0	2.94	28	75
			C8	15.4	4.84	26	72
			D8	84.7	1.33	29	64

TABLE 2--Continued  
Stress-Rupture Results

Temp. (°F)	Stress (psi)	Code	Rupture Time (hr)	Min. Creep Rate (in./in./1000 hr)	Elongation (%)	Reduction of Area (%)
900	80,000	A1	102.5	.67	28	73
		B1	65.8	1.25	31	73
		C1	206.0	.46	36	71
		D1	445.5	.245	33	59
		AR*	50.3	.927	18	68
		BR	58.5	1.18	20	71
		CR	74.0	.90	21	67
		DR	281.0	.317	27	59
	75,000	A6	419.2	.133	29	67
		B6	342.0	.246	31	67
		C6	347.0	.262	36	68
		D6	732.6	.128	24	52
	70,000	A9	1100.	.0488	25	36
		B9	1120.8	.0547	22	48
		C9	1352.0	.0492	24	34
		D9	1193.4	.065	25	46
1000	50,000	A4	104.7	.528	13	22
		B4	101.5	.513	17	22
		C4	97.9	.835	25	32
		D4	108.4	1.01	32	46
	45,000	A3	182.4	.197	10	12
		B3	192.0	.247	13	15
		C3	176.0	.328	14	15
		D3	188.3	.354	27	33

\*Specimens initially tested in relaxation at 900° F - 80,000 psi for 500 hours.

The effect of cold work on the rupture strength of the steel varied depending on the temperature (Fig. 1). The 100-hour rupture strength at 700° F (370° C) was greatly enhanced by prior cold work, the improvement increasing with larger amounts of cold work. At 800° and 900° F (425° and 480° C) the rupture strength was generally lowered by critical amounts of cold work (8 and 15 %), while beyond the critical values increased strength was obtained. The lowest 100-hour rupture

strength was exhibited by the 8 % cold-worked material. The possible severity of this detrimental effect is shown by the reduction in rupture time to approximately 15 % of its original value at 800° F (425° C) and 100,000 psi, Table 2. At 1000° F (540° C) there was no apparent influence of cold work on rupture strength for tests of all durations as shown in Fig. 1. The variation of the 1000-hour rupture strength due to prior deformation, presented in Fig. 2, was similar to that of the 100-hour rupture strength at 800° F (425° C); however, at 900° F (480° C) a small increase in the 1000-hour strength with increasing cold work indicated a behavior differing from that of the shorter time strength. Apparently the longer test time at 900° F (480° C) alleviated the influence of the cold deformation.

The effect of cold work on creep strength, (stress for creep rate of 0.5 in./in./1000 hr) was different at the various temperatures (Fig. 3). An improvement in creep strength with increasing cold work was obtained at 700° F (370° C). At 800° and 900° F (425° and 480° C) the creep strength was lowered by 8 and 15 % cold reduction; a reduction of 39 % raised the strength. A general decrease in creep strength with larger amounts of cold work was indicated at 1000° F (540° C).

The decrease in 100-hour rupture strength with increase in temperature, shown in Fig. 4, is essentially linear to 900° F (480° C); beyond this temperature the rate of weakening is greater. The loss of strength due to temperature appeared to be slightly greater for the 39 % cold-worked material than for the less-cold-worked material.

There was no apparent effect of cold work on elongation at fracture except at 1000° F (540° C) where increasing prior deformation produced higher elongations (Fig. 5). The general level of elongation values was raised with increase in temperature to 900° F (480° C); at 1000° F (540° C) a downward trend was indicated. Variation of initial stress (or time of test) had no apparent effect on elongations at fracture at 700° and 800° F (370° and 425° C). However, at 900° F (480° C) higher elongations were generally obtained at the two intermediate stress levels, and at 1000° F (540° C) smaller elongations were produced by the lower stresses.

No change in reduction of area due to cold work was observed at 700°, 800°, and 900° F (370°, 425°, and 480° C) except for the 39 % reduced material which generally showed lower values (Fig. 6). At 1000° F (540° C) reduction of area increased with increase in deformation. There was no significant effect of stress at 700° and 800° F (370° and 425° C); however, at 900° and 1000° F (480° and 540° C) reduction of area decreased with decreasing stress. The general level of reduction of area values was lower at 1000° F (540° C) than at the other temperatures.

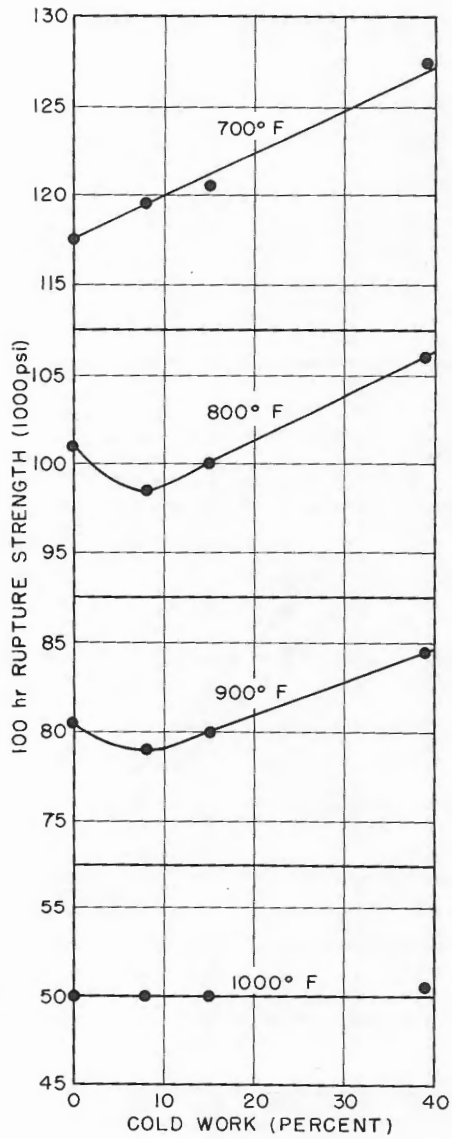


Fig. 1 - Effect of cold work on 100-hr rupture strength at 700°, 800°, 900°, and 1000°F

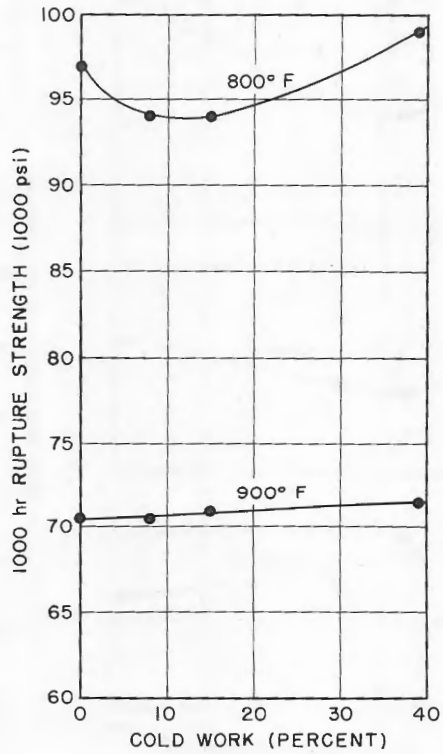


Fig. 2 - Effect of cold work on 1000-hr rupture strength at 800° and 900°F

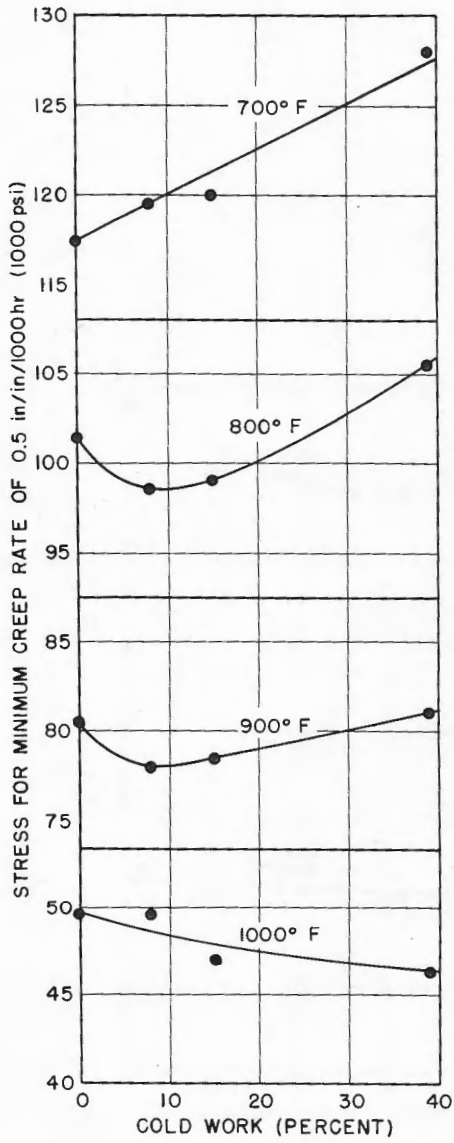


Fig. 3 - Effect of cold work on stress for creep rate of 0.5 in./in./1000 hr at 700°, 800°, 900°, and 1000°F

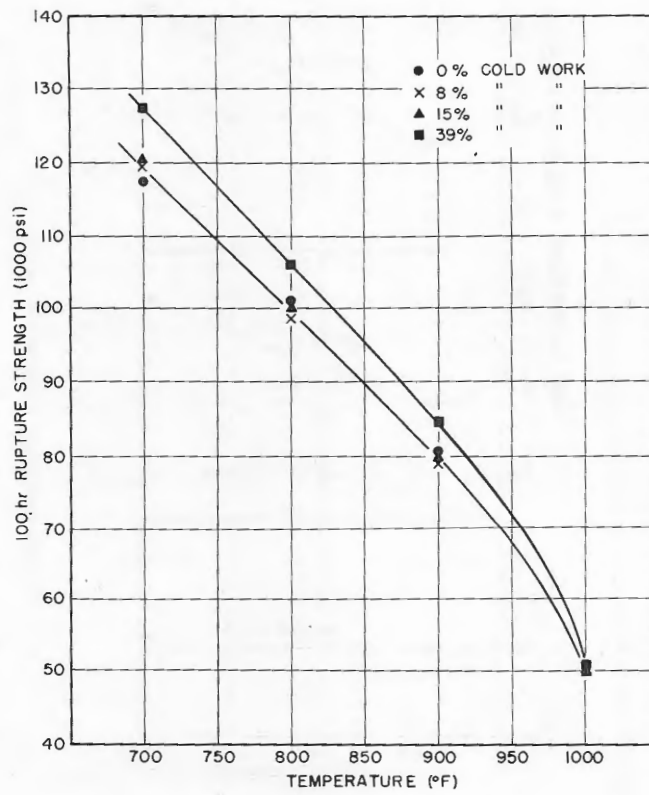


Fig. 4 - Effect of temperature on 100-hr rupture strength

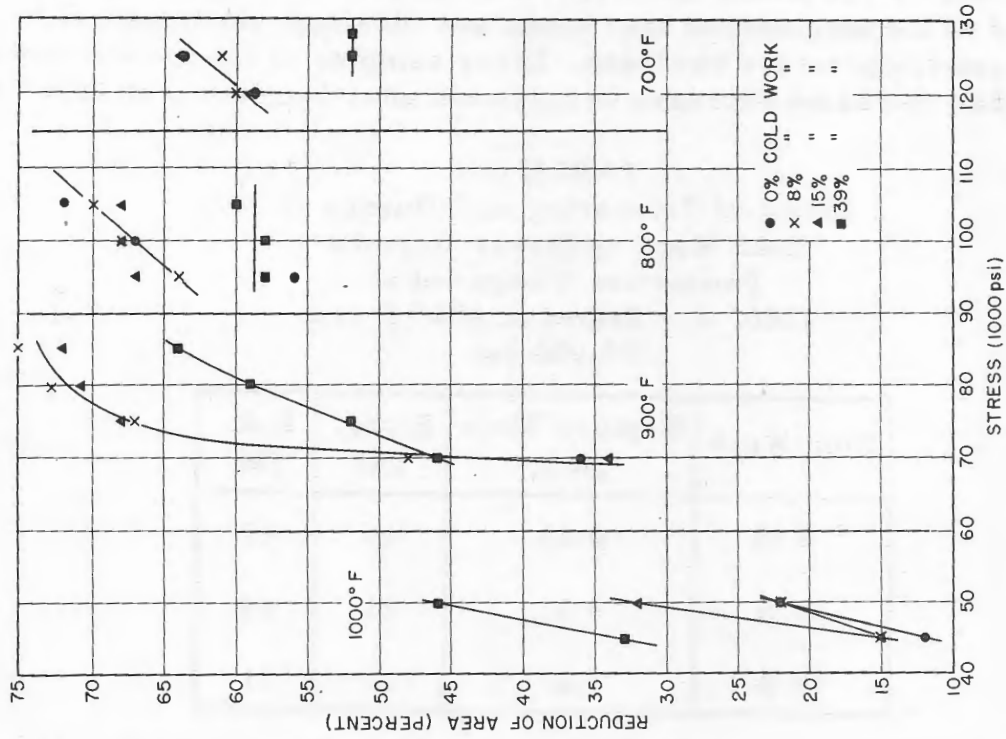


Fig. 6 - Influence of cold work and stress on reduction of area at 700°, 800°, 900°, and 1000°F

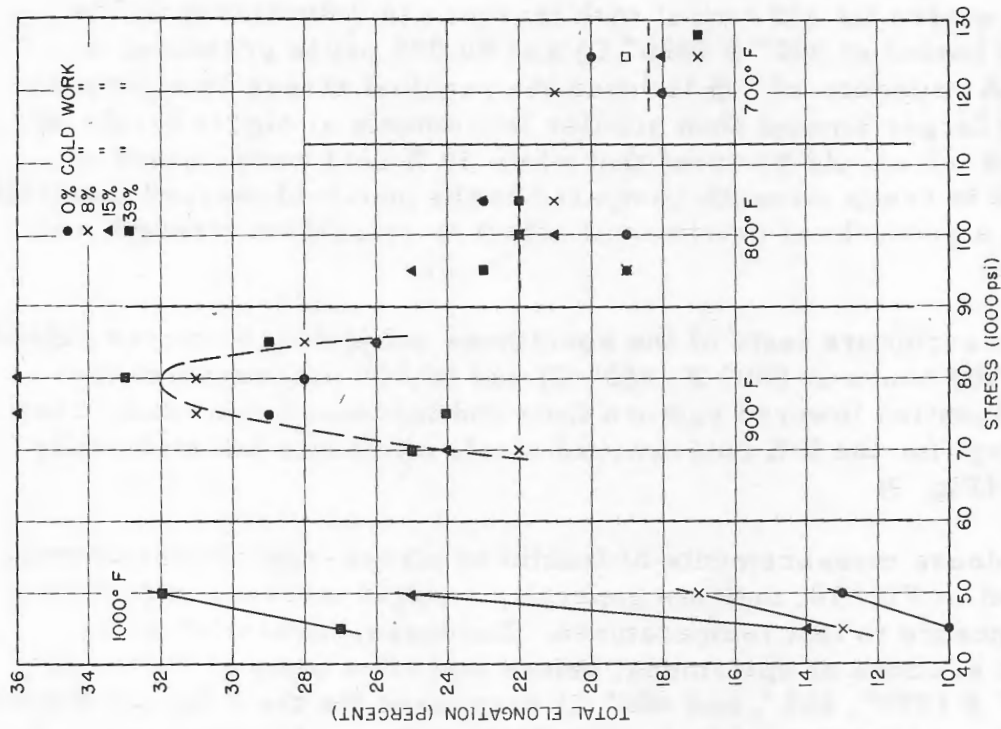


Fig. 5 - Influence of cold work and stress on elongation at fracture at 700°, 800°, 900°, and 1000°F

Tempering the cold worked steel at 1200° F prior to testing in stress-rupture did not eliminate the influence of cold work on the properties at 800° F (Table 3). However, a lower level of rupture times compared to the untempered specimens was obtained, accompanied by lower room-temperature hardness. Other samples of the worked steel did not show the same decrease in hardness after tempering at 1200° F.

TABLE 3  
Effect of Tempering on Influence of  
Cold Work on Stress-Rupture  
Properties Tempered at  
1200° F - Tested at 800° F and  
100,000 psi

Cold Work	Rupture Time (hr)	Elong. (%)	R.A. (%)
8 %	0.05	19	65
15 %	0.5	21	64
39 %	1.2	20	61

In stress relaxation tests, increased cold work produced greater relaxation at 900° F (480° C), as shown in Fig. 7. The decrease in residual stress (at 500 hours) with increase in deformation of the material loaded at 900° F (480° C) and 80,000 psi is presented in Fig. 8. A reduction of 8 % lowered the residual stress by a proportionately larger amount than similar increments at higher levels of cold work. It should be noted that while 39 % cold work caused no decrease in creep strength compared to the non-cold-worked material (Fig. 3), a pronounced detrimental effect on relaxation strength was developed.

Stress-rupture tests of the specimens subjected to stress relaxation for 500 hours at 900° F (480° C) and 80,000 psi, revealed that prior relaxation lowered rupture time and increased minimum creep rate except for the 8 % cold-worked steel, which was not materially affected (Fig. 9).

Hardness measurements of fractured stress-rupture specimens, presented in Fig. 10, indicate generally a slight increase in hardness after exposure to test temperatures. Hardness, measured on the threaded sections of specimens, before and after tests at 700°, 800°, and 900° F (370°, 425°, and 480° C) decreased for the 8 % cold-worked material and increased for the materials with greater cold work. A general increase in hardness with increasing prior cold work was observed in the gage sections after tests at these temperatures.

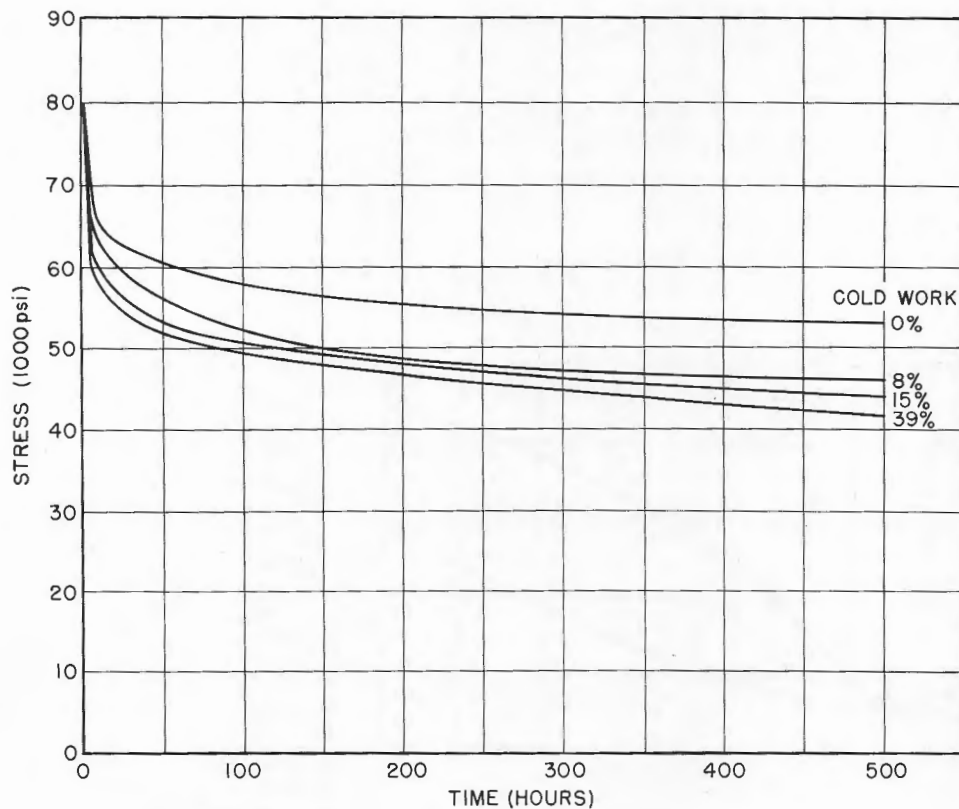


Fig. 7 - Stress relaxation curves of cold worked Cr-Mo steel at 900°F and 80,000 psi initial stress

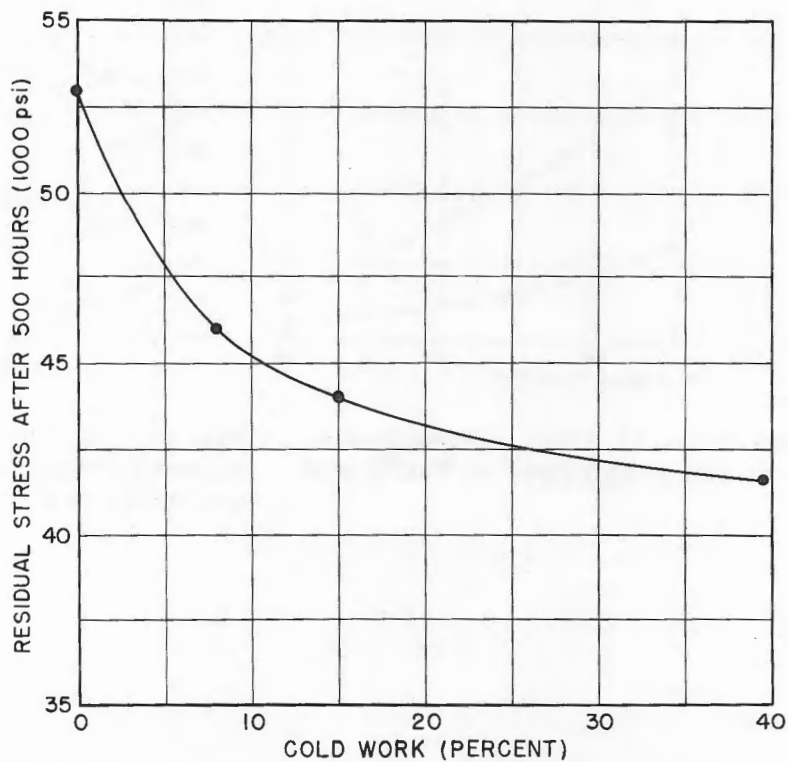


Fig. 8 - Effect of cold work on relaxation at 900°F and 80,000 psi initial stress

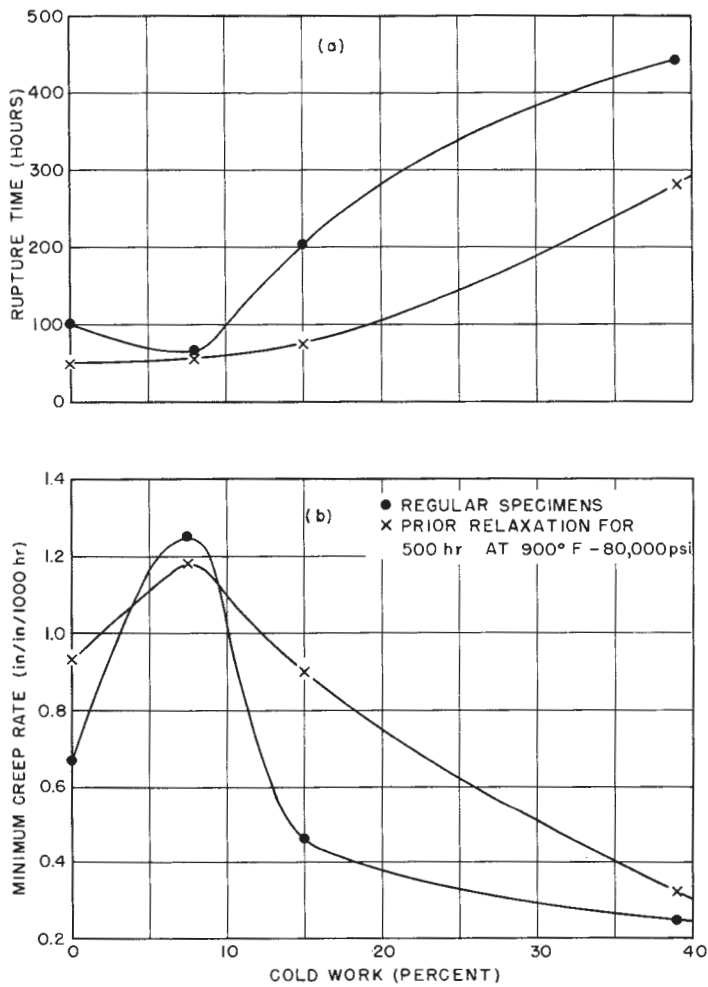


Fig. 9 - Influence of prior relaxation on rupture time and creep rate at 900°F and 80,000 psi

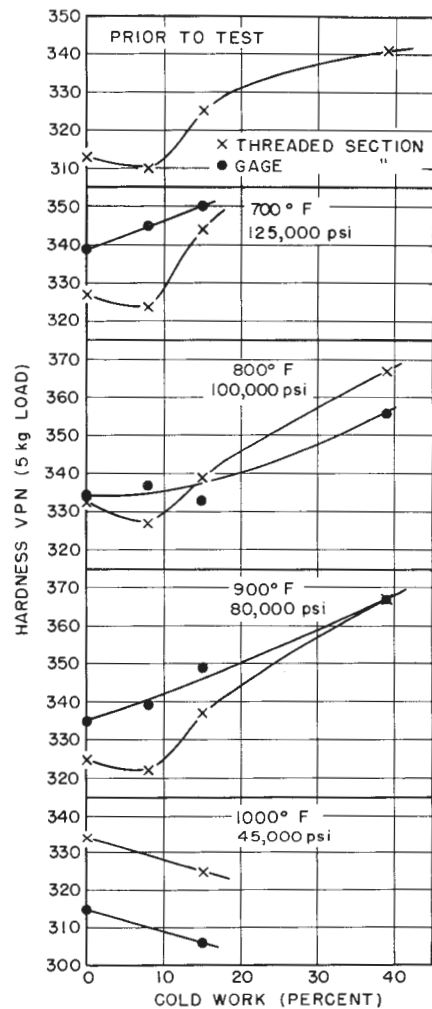


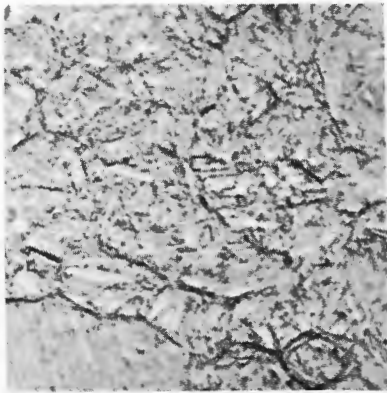
Fig. 10 - Hardness of stress-rupture specimens prior to and after tests

Fractured specimens which had been tested at stresses above 70,000 psi at 900° F (480° C) and below displayed necking; those tested below this stress level showed intergranular fractures without necking, except for a 39 % cold-worked specimen under 50,000 psi, which exhibited a slight neck. At 70,000 psi with a temperature of 900° F (480° C), either type of fracture occurred.

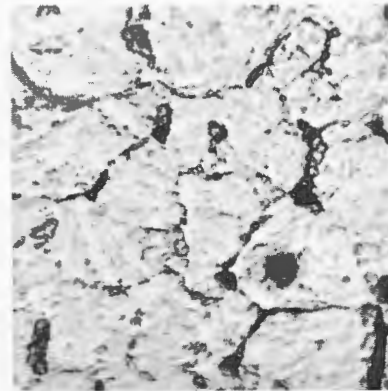
Metallographic examination of fractured specimens (Fig. 11) did not reveal any evidence of recrystallization. Coalescence of cementite was observed in specimens tested at the higher temperatures, particularly in the highly cold-worked material. Intergranular oxidation was evident in the gage sections of specimens tested at 1000° F (540° C).

The lowering of rupture strength and creep resistance at 800° and 900° F (425° and 480° C) by small amounts of prior cold deformation, is explainable on the basis of the Bauschinger effect. The Bauschinger effect (13) is concerned with the change in mechanical properties on reversing the direction of straining. Straining, in either tension or compression, raises the ability of the metal to withstand further stressing of the same-kind and lowers its strength to stresses of the opposite kind. Work-softening at room temperature, believed to be of this type, was observed in this study, Table 1. Since the final straining was in the same direction (tensile) with all the specimens, the differences noted may be related to differences in the initial stress state of the 8 and 15 % cold-worked material compared to the 39 % cold-worked material. According to Baldwin (14) the residual-stress state resulting from nonpenetrating rolling (slight reductions) is different from that produced by penetrating (heavy) rolling. Light rolling produces a compressive stress at the rolled surface and a tensile stress in the interior whereas heavy rolling produces a tensile stress at the rolled surface and a compressive stress in the interior, all acting in the rolling direction. Presumably the stress condition (with slight adjustments) remains after the machining of specimens. The decrease in room-temperature yield strength of the material by 8 % cold work may be attributed to the Bauschinger effect. This loss in strength due to a critical degree of deformation appears to carry to elevated temperatures, in this case to 900° F (480° C), and is evident in the creep and rupture strengths. This seems reasonable, since it has been reported (15) that heating a 0.28 % carbon steel at 400° C (750° F) for 8 hours after prestraining did not eliminate the work-softening effect. The absence of work-softening effects in our tests at 700° F (370° C) may be due to additional cold-working of the material (5 - 6 % elongation) upon application of the test load.

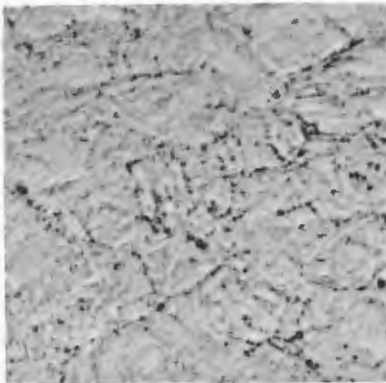
On the basis of hardness measurements, Fig. 10, softening by the 8 % deformation persists even after prolonged holding at 900° F (480° C); however, straining appears to remove the softening. It should be noted that the hardness changes in the critical region were small. A similar



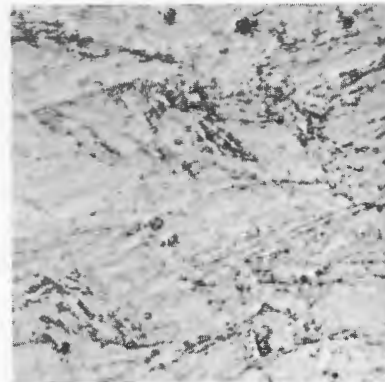
Unworked  
700<sup>0</sup>F - 1.5 hrs  
to rupture



Unworked  
1000<sup>0</sup>F - 182.4 hrs  
to rupture



Unworked  
900<sup>0</sup>F - 102.5 hrs  
to rupture



15% cold worked  
900<sup>0</sup>F - 206.0 hrs  
to rupture

Fig. 11 - Photomicrographs of fractured specimens (1/4" from fracture). Zephiran chloride, ether, picric acid etchant (1000 X).

behavior, work-softening of a quenched and tempered steel, was reported by Polakowski (15), who presented the explanation that the steel in the quenched and tempered condition contains considerable internal stresses and consequently behaves like metals in which the internal stresses have been produced by cold work. It should therefore soften during cold-work. However, the method of measuring this softness, indentation hardness with its complex mechanics, may be a factor in the observation. Microstructure has been shown by Wilson (16) to influence this effect; a sorbitic structure showed the largest softening whereas a spheroidized structure displayed no softening. The work-softening effect observed in this study is probably based on the residual macrostresses and related microstresses induced by cold work.

The effect of cold work (39 %) on relaxation strength and creep strength was found to be different at 900° F (480° C); the relaxation strength was lowered whereas the creep strength was slightly improved. This indicates that the steady-state creep, used as measure of creep strength, is not the controlling factor in relaxation. The first stage of creep is probably of more significance in the estimation of relaxation behavior, although no evidence of this was apparent in this study.

## SUMMARY AND CONCLUSIONS

The influence of cold work on creep, rupture, and relaxation properties of a quenched and tempered chromium-molybdenum steel was investigated. The material with 0, 8, 15, and 39 % cold reduction was tested in stress-rupture at 700°, 800°, 900°, and 1000° F (370°, 425°, 480°, and 540° C) in the stress range of 45,000 to 128,000 psi. Relaxation tests were conducted at 900° F (480° C) with an initial stress of 80,000 psi for 500 hours followed by stress-rupture tests of the relaxed specimens. Room temperature tensile properties of the unworked and worked steel were determined.

From the results of this study, the following conclusions may be drawn:

1. The effect of cold work on the creep and rupture properties of chromium-molybdenum steel varies, depending on temperature and degree of deformation. Relaxation is affected in a different manner from creep.

2. Creep strength (stress to produce a creep rate of 0.5 in./in./1000 hr) and 100-hour rupture strength increase with increasing cold work at 700° F (370° C).

3. Creep strength and 100-hour rupture strengths at 800° and 900° F (425° and 480° C) are lowered by small amounts of cold work, 8 and 15 %, but are improved by 39 % cold work. The behavior is explainable on the basis of the Bauschinger effect. The effect of cold work at 900° F (480° C) is less in long time tests (1000 hours).

4. At 1000° F (540° C) no effect of cold work is apparent on rupture strength; however, a decrease in creep strength is indicated with increasing cold work.

5. There is no apparent effect of cold deformation on elongation at fracture except at 1000° F (540° C), where increasing prior deformation produces higher elongation values. Reduction of area is also unaffected at 700°, 800°, and 900° F (370°, 425°, and 480° C) except for the 39 % reduced material, which is generally lower. At 1000° F (540° C) reduction of area increases with increase in deformation.

6. A decrease of residual stress in relaxation tests is obtained with increasing cold work at 900° F (480° C) and 80,000 psi initial stress.

7. Room-temperature tensile strength, 0.2 % yield strength, and hardness are lowered slightly by 8 % cold work; with greater cold work (15 and 39 %), increases are obtained.

#### ACKNOWLEDGMENT

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