NACA TN 2586

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2586

FUNDAMENTAL EFFECTS OF COLD-WORK ON SOME COBALT-CHROMIUM-NICKEL-IRON BASE CREEP-RESISTANT ALLOYS

By D. N. Frey, J. W. Freeman, and A. E. White
University of Michigan

NACA
Washington
January 1952

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2586

FUNDAMENTAL EFFECTS OF COLD-WORK ON SOME COBALT-CHROMIUM-NICKEL-IRON BASE CREEP-RESISTANT ALLOYS

By D. N. Frey, J. W. Freeman, and A. E. White

SUMMARY

The influence of cold-working on the creep properties of an alloy containing 20 percent cobalt, 20 percent chromium, 20 percent nickel, and the balance iron and on the same alloy modified by small additions of tungsten alone or tungsten, molybdenum, and columbium in combination was studied. It was concluded that the effects of cold-working on creep resistance were the same for all the alloys studied. This was from the standpoint of the temperature range over which cold-working could be expected to be beneficial (temperatures up to 1600° F) and also the maximum amount of cold-working which could be used to improve creep properties at about 1200° F (between 15 and 40 percent). These conclusions were reached in part by studying the creep properties and also in part by studying internal stress relaxation at the test temperatures which previous work had shown to be the controlling factor in the response of such alloys to cold-working.

INTRODUCTION

This report is the fifth of a series concerned with the fundamental factors influencing the creep properties of creep-resistant alloys for use in aircraft propulsion systems. Previous articles have dealt with the influence of chemical composition, precipitation, and cold-working on high-temperature properties of such alloys (see references 1, 2, 3, and 4).

One of the previous investigations (see reference 1) showed fairly conclusively that the improvement in creep properties to be had by cold-working low-carbon N-155 alloy was the result of the introduction of internal stresses - such stresses acting to broaden X-ray diffraction lines. Further it was found that increasing degrees of cold reduction resulted in improved creep resistance up to that amount which caused significant X-ray line sharpening, which is interpreted as internal stress relaxation[1], to occur at the creep test temperature. From this it was also reasonable to expect that the maximum temperature at which cold-working can be expected to improve creep resistance will be the maximum temperature at which appreciable internal stresses (X-ray line breadths) are retained during test or service. It also leads to a redefinition of "cold-working" as working at any temperature for which no appreciable internal stress relaxation occurs during the working operation or during the cool down after such working. The temperature dependence of the influence of "cold-working" on creep resistance was checked and found to agree with this view.

One unanswered question which immediately presented itself was how the response to cold-working might vary in a fundamental sense with chemical composition. In view of the foregoing results it appeared that this could be done by studying the influence of chemical composition on the creep properties after cold-working and on the internal-stress-relaxation characteristics (line sharpening) - the latter to be used as a fundamental explanation for the former results. The purpose of this investigation therefore was to measure the influence of various minor additions (tungsten and tungsten, molybdenum, and columbium in combination) on the creep properties and on the line sharpening or internal-stress-relaxation characteristics of cold-worked austenitic alloys containing 20 percent cobalt, 20 percent chromium, 20 percent nickel, and the balance iron. This base and the minor additions were chosen for two reasons. First, the base and the minor additions were felt to be representative of austenitic alloys in use commercially. Second, creep and rupture data on the alloys, in the solution-treated state, were available which showed rather large variation in high-temperature strength due to the small composition differences. It was initially believed that large variation in creep or rupture resistance might also mean variation in resistance to internal stress relaxation.

This investigation was conducted at the Engineering Research Institute of the University of Michigan under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

---

[1] The factors which might cause diffraction line broadening by cold-working were examined and reported in reference 1. The conclusion was reached that such broadening was due to the introduction of internal stresses, and, further, the line sharpening which occurred at elevated temperatures was due to internal relaxation of these stresses.

TEST MATERIALS

The alloys used in this investigation were all made as 12-pound induction furnace heats with the following analyses:

Heat	Chemical composition (percent) (1)									
	C	N	Mn	Si	Cr	Ni	Co	Mo	W	Cb
43	0.12	(0.12)	1.7	0.50	(20)	(20)	(20)	0.09	0	0
86	.17	.13	1.67	.69	20.57	20.8	18.77	(0)	0	(0)
88	0.19	(0.12)	(1.7)	(0.5)	(20)	(20)	(20)	(0)	1.89	(0)
91	0.17	(0.12)	(1.7)	(0.5)	(20)	(20)	(20)	(0)	3.96	(0)
92	0.18	(0.12)	(1.7)	(0.5)	(20)	(20)	(20)	(0)	5.99	(0)
106	.15	(.12)	(1.7)	(.5)	(20)	(20)	(20)	(0)	5.60	(0)
12	0.16	0.14	1.61	0.67	21.04	18.39	22.00	3.03	2.01	1.15
95	(.12)	(.12)	(1.7)	(.5)	(20)	(20)	(20)	(3)	(2)	(1)

[1] Figures not in parenthesis are by actual analysis; figures in parenthesis represent aim or nominal contents.

Five basically different alloys appear above: The base alloy (heats 43 and 86), the base alloy plus 2 percent tungsten (heat 88), the base alloy plus 4 percent tungsten (heat 91), the base alloy plus 6 percent tungsten (heats 92 and 106), and the base alloy plus 3 percent molybdenum, 2 percent tungsten, and 1 percent columbium (heats 12 and 95). This last alloy corresponds to the commercial alloy known as low-carbon N-155. Duplicate heats of the same type appear where insufficient stock was available from any one heat for all the needed specimens.

The heats were cast as 9-inch-long tapered (2- to $2\frac{1}{2}$-in.-square) big-end-up ingots, with $2\frac{3}{4}$-inch diameter by 2-inch tops. For purposes of this investigation, portions of the ingots were forged over the temperature range of 2200° to 1850° F to approximately 3/4-inch-square bars (heats 86, 88, 91, 92, and 95) and to approximately 1/2-inch round bars (heats 12, 43, 88, 91, and 106). Ten to twenty reheats were used for all ingots with reductions of the order of 15 percent between reheats.

Forging in all cases was done under a 400-pound hammer, with flat-faced dies being used for the square stock and swages for finishing the round bars.

Both the square bars (to be used for cold-rolling) and the round bars (to be used for creep testing of solution-treated stock) were solution-treated 1 hour at 2200° F and water-quenched, with the square bars having been machined to exactly 3/4 inch square before solution treatment. The solution treatment was used to eliminate, to the fullest possible extent, prior-processing history in the various alloys and bar forms so that subsequent differences in behavior would be due to the differences in composition. The efficiency of this high-temperature solution treatment in removing prior-processing variation as a variable has been considered elsewhere (see reference 2).

EXPERIMENTAL PROCEDURE

The solution-treated 3/4-inch-square bars of heats 86, 88, 91, 92, and 95 described above, were first reduced 15 and 40 percent in cross section at 80° F. These reductions were carried out in a two-high, non-reversing mill with 5-inch rolls. Sufficient stock of all five heats was prepared in each reduction to give samples for X-ray diffraction analysis and creep test specimens.

The creep properties of the two cold-rolled conditions of heats 86, 88, 91, 92, and 95 were surveyed by creep testing to rupture under a stress of 40,000 psi at 1200° F. This stress corresponded roughly to the 1000-hour rupture strength of the strongest alloys tested. The estimated error in the creep rates so determined was ±40 percent. The procedure for carrying out these creep tests was exactly the same as that covered in reference 1.

In addition to the above tests the creep properties of the as-solution-treated alloys (heats 43, 88, 91, 106, and 12) were surveyed at the same stress and temperature as above. The test specimens for this were made from the 1/2-inch round bars.

For the line-sharpening studies, samples of each of the heats, cold-rolled 15 percent, were then annealed for 1000 hours at 1200° F, 10, 100, and 1000 hours at 1400° F, and 30 hours at 1600° F. These times and temperatures were chosen to survey the line-sharpening characteristics of the various alloys with a minimum of testing. Also samples of each of the above heats cold-rolled 40 percent were annealed 100 and 1000 hours at 1200° F. Following this annealing, the half-height width of the [220] diffraction line (taken with chromium K radiation and having a Bragg angle θ equal to 67°) of each of the annealed conditions plus the as-rolled conditions was determined by a procedure previously described in reference 1. The estimated error determination of the line widths by this procedure was ±10 percent. This procedure assumes that the width of the [220] line of the as-solution-treated samples represents broadening due to all causes except the residual stresses from cold-working. The widths of the rolled and rolled-and-annealed samples were corrected for this broadening by the method of Warren and Biscoe (see reference 5).

RESULTS AND DISCUSSION

Figure 1 shows the variation, with amount of cold reduction, of the creep rate of the various alloys under 40,000 psi at 1200° F. Figure 2, essentially a cross plot of the data shown in figure 3, shows more clearly than figure 3 the effect of tungsten on the creep rate under 40,000 psi at 1200° F of the base alloy cold reduced 0, 15, and 40 percent. Also shown, as individual points, are similar data for the alloy containing 3 percent molybdenum, 2 percent tungsten, and 1 percent columbium which corresponds to low-carbon N-155. Figure 3 shows the effect of annealing on the [220] line width of the various solution-treated alloys when cold reduced 15 percent, while figure 4 shows the effect of annealing at 1200° F on the [220] line width of the same alloys cold reduced 40 percent.

Figure 1 indicates that the optimum amount of cold reduction as far as creep resistance at 1200° F is concerned was between 15 and 40 percent and was the same for all the alloys. The curves shown in figure 1 are drawn with a minimum, although the data do not completely justify it. However, the results of Zschokke (see reference 6) on another austenitic alloy with a greater range of reduction showed an actual minimum and thus the curves are drawn as shown.

Figure 2 shows that the relative order of merit of the alloys as far as creep at 40,000 psi and 1200° F is concerned remains the same whether they are cold-worked or not. There are reasons to suspect that the relative strengths of the alloys might be different at different stress levels but that again this order would be unchanged by cold-working. The important point then is that relative improvement of the alloys considered herein is not possible by cold-working.

Figure 3 indicates that, within the experimental error, the [220] line widths resulting from 15-percent cold reduction and after subsequent annealing were the same for all the alloys. Particularly important to note is the absence of appreciable line sharpening at 1200° F. All this can be interpreted to mean that, for cold reductions of 15 percent, all the alloys are equally stable as far as internal stress relaxation is concerned at 1200° F and exhibit the same tendency for such stress relaxation at higher temperatures.

Figure 4 shows that within the experimental error all the alloys, cold reduced 40 percent, behaved similarly in regard to line sharpening at 1200° F. Appreciable sharpening occurred in all the alloys. The base alloy possibly underwent slightly more line sharpening (internal stress relaxation) than the others, although the point lies almost within the experimental error. The extent of the sharpening at 1200° F for all the alloys was such as to reduce the line width to a value comparable with that for the alloys reduced 15 percent (the latter reduction either with or without an anneal at 1200° F). This would indicate on the basis of a linear correlation between diffraction-line widths and logarithm of creep rate at 1200° F for any given composition and any given stress (see reference 1) that the creep properties at 1200° F of any one of the alloys cold reduced 40 percent would be no better than those of the same alloy cold reduced 15 percent. This was exactly what was observed in the creep testing covered above. In another sense the results in figures 3 and 4 indicate that for all the alloys considered herein, cold reductions of the order of 40 percent were sufficient to lower the minimum temperature for significant internal stress relaxation to 1200° F.

The results shown in figures 3 and 4 further indicate that significant improvement in creep resistance over the solution-treated state by cold-working can be expected up to temperatures of almost 1500° F. This statement is made on the basis that some internal stress is retained up to such a temperature, and, further, that the results in both this investigation and reference 1 point to the fact that it is such internal stresses which improve the creep properties.

The similarity of all the alloy compositions studied with regard to line sharpening or internal stress relaxation was unexpected on the basis of rupture tests and some creep tests on the same materials (see reference 2). In these tests uniform and marked improvement in strength at 1200° F was found as the result of the addition of tungsten up to 6 percent or 3 percent molybdenum, 2 percent tungsten, and 1 percent columbium in combination. It was assumed that improvement in creep resistance, for example, would result in increased resistance to internal stress relaxation. However, it now appears that the internal-stress-relaxation characteristics of such alloys as those considered herein are more a function of the gross composition of the alloy. Significant variation in such recovery characteristics as line sharpening is known to exist with gross-composition changes.

CONCLUSIONS

The following conclusions were drawn from an investigation of the influence of cold-working on the creep properties of an alloy containing 20 percent cobalt, 20 percent chromium, 20 percent nickel, and the balance iron and on the same alloy modified by small additions of tungsten alone or tungsten, molybdenum, and columbium in combination:

1. The amount of cold reduction for maximum creep resistance at 1200° F was the same - approximately 25 percent - for the base alloy and the same alloy modified by additions of tungsten up to 6 percent or 3 percent molybdenum, 2 percent tungsten, and 1 percent columbium in combination.

2. Because the internal stresses represented by diffraction line widths control creep properties, it appeared from studies of diffraction line sharpening that the amount of cold-working for maximum creep resistance at 1200° F was the same for all the above alloys - between 15 and 40 percent reduction in agreement with the above finding. Reduction of 40 percent or greater resulted in appreciable line sharpening or internal stress relaxation at 1200° F of all the alloys, such relaxation acting to lower the creep resistance to that for reductions of 15 percent.

3. The cold-worked base alloy and the cold-worked base alloy with the above-mentioned minor additions exhibited the same line sharpening or internal-stress-relaxation characteristics upon annealing in the range 1200° to 1600° F for time periods ranging up to 1000 hours.

4. It appeared from those line width studies that the maximum temperature at which cold-working can be expected to improve creep resistance, for time periods up to 1000 hours, is the same for all the alloys - approximately 1600° F.

5. The similarity in line-sharpening characteristics and the similarity in optimum cold reduction for creep resistance at 1200° F for all the alloys covered herein indicated that changes of internal-stress-relaxation characteristics of such alloys will be dependent upon rather large changes in chemical composition rather than the minor changes in composition studied herein.

University of Michigan
Ann Arbor, Mich., April 1, 1951

REFERENCES

1. Frey, D. N., Freeman, J. W., and White, A. E.: Fundamental Effects of Cold-Working on Creep Properties of Low-Carbon N-155 Alloy. NACA TN 2472, 1951.

2. Reynolds, E. E., Freeman, J. W., and White, A. E.: Investigation of Influence of Chemical Composition on Forged Modified Low-Carbon N-155 Alloys in Solution-Treated and Aged Condition as Related to Rupture Properties at 1200° F. NACA TN 2449, 1951.

3. Frey, D. N., Freeman, J. W., and White, A. E.: Fundamental Effects of Aging on Creep Properties of Solution-Treated Low-Carbon N-155 Alloy. NACA Rep. 1001, 1950. (Formerly NACA TN 1940.)

4. Frey, D. N., Freeman, J. W., and White, A. E.: Fundamental Aging Effects Influencing High-Temperature Properties of Solution-Treated Inconel X. NACA TN 2385, 1951.

5. Warren, B. E., and Biscoe, J.: The Structure of Silica Glass by X-Ray Diffraction Studies. Jour. Am. Ceramic Soc., vol. 21, 1938, pp. 49-54.

6. Zschokke, H. R., and Niehus, K. H.: Requirements of Steel for Gas Turbines. Jour. Iron and Steel Inst., vol. 156, pt. 2, June 1947, pp. 271-283.

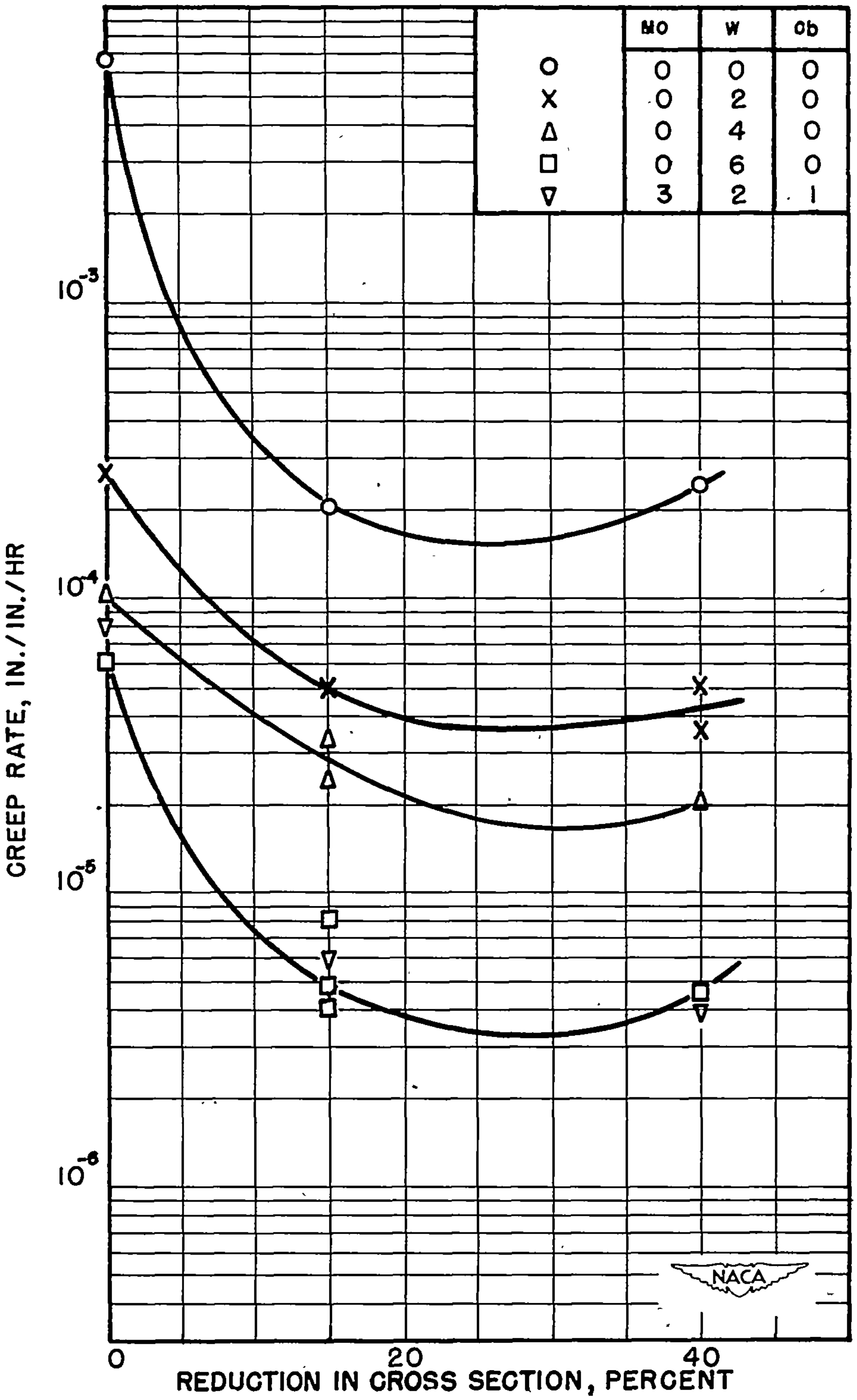

Figure 1.- Effect of reduction at 80° F on creep rate of solution-treated base alloy, modified as indicated, under 40,000 psi at 1200° F.

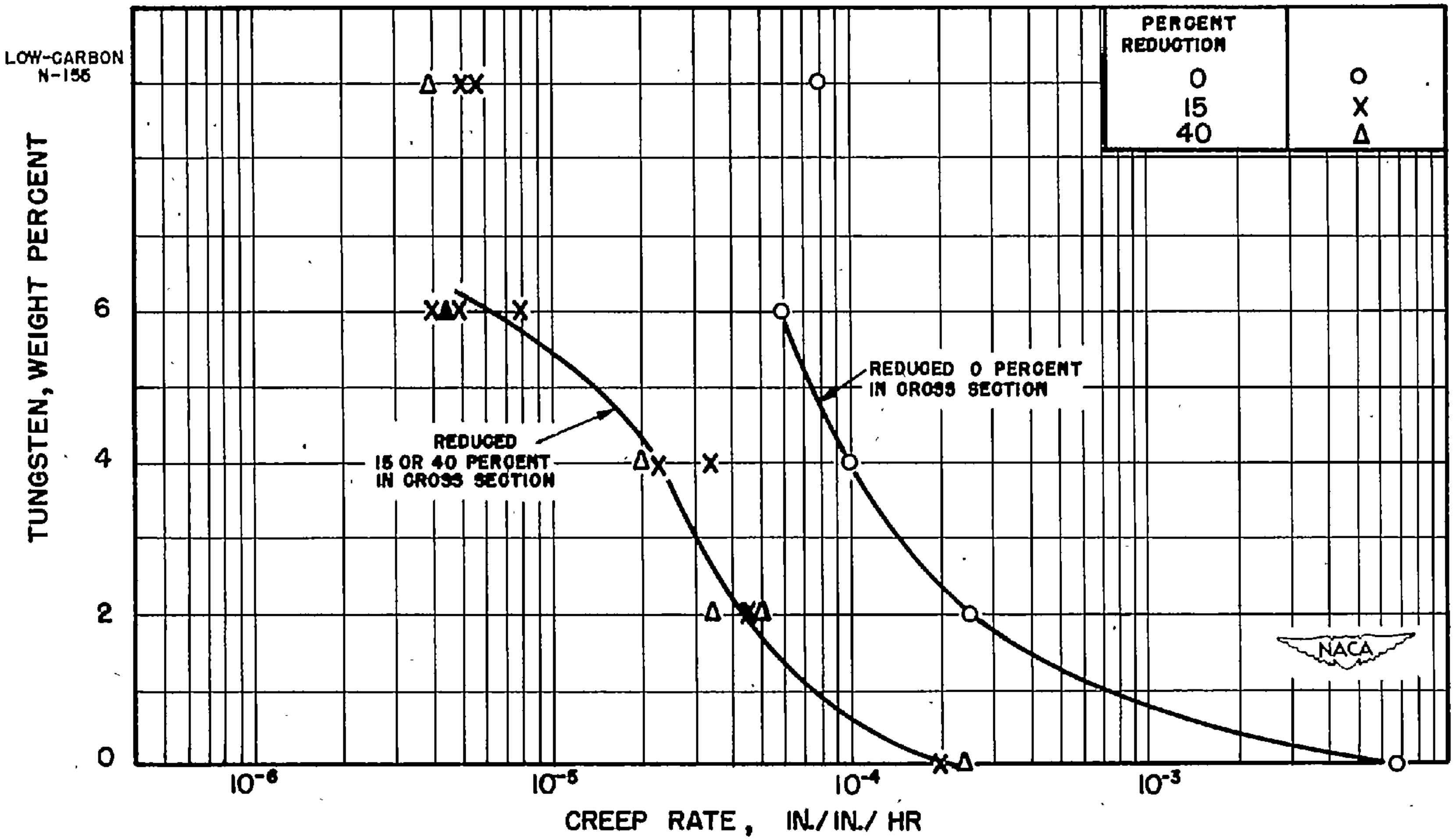

Figure 2.- Effect of tungsten on creep rate of solution-treated and cold-reduced base alloy under 40,000 psi at 1200° F.

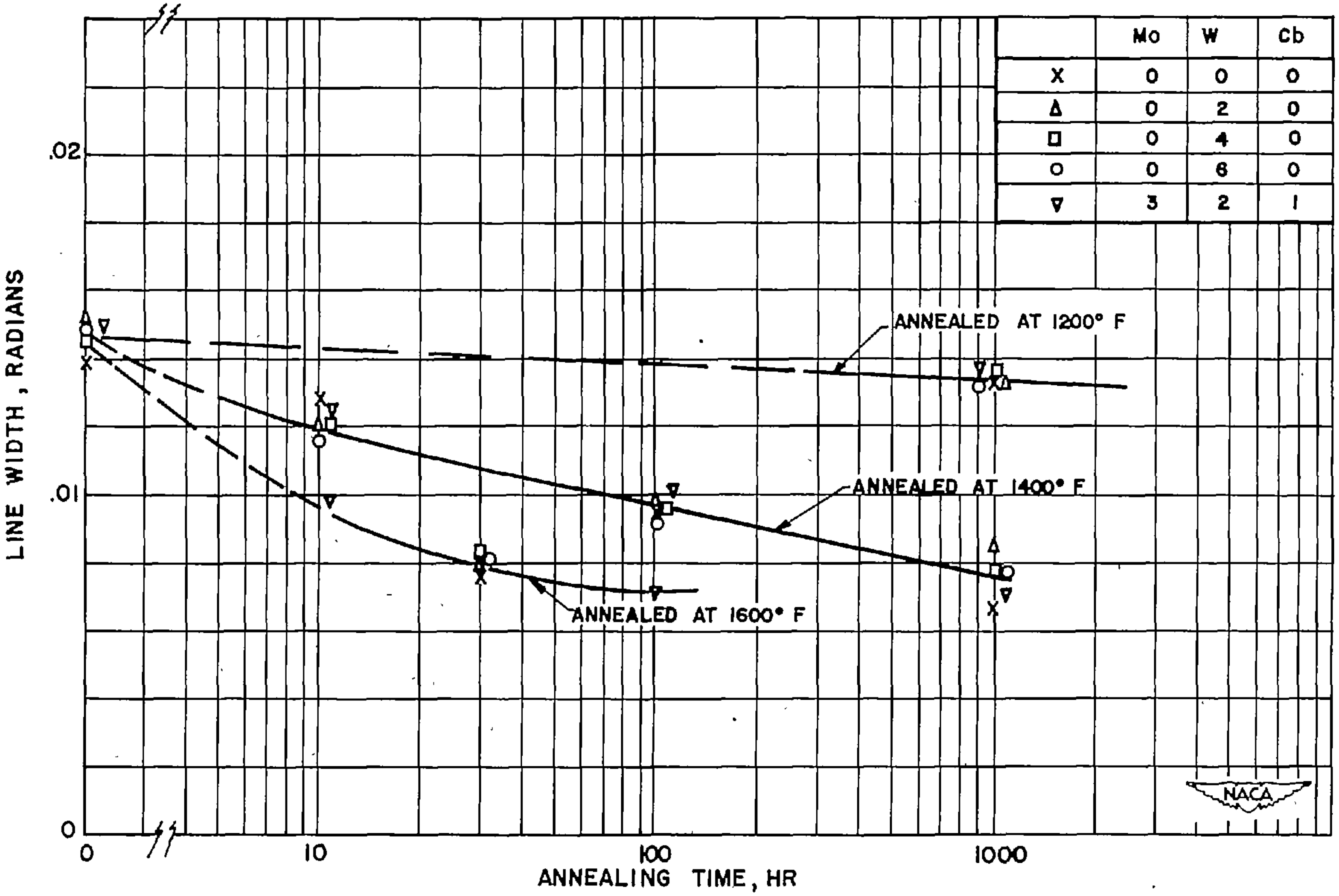

Figure 3.- Effect of annealing on [220] line width of base alloy, modified as indicated, solution-treated 1 hour at 2200° F and reduced 15 percent at 80° F.

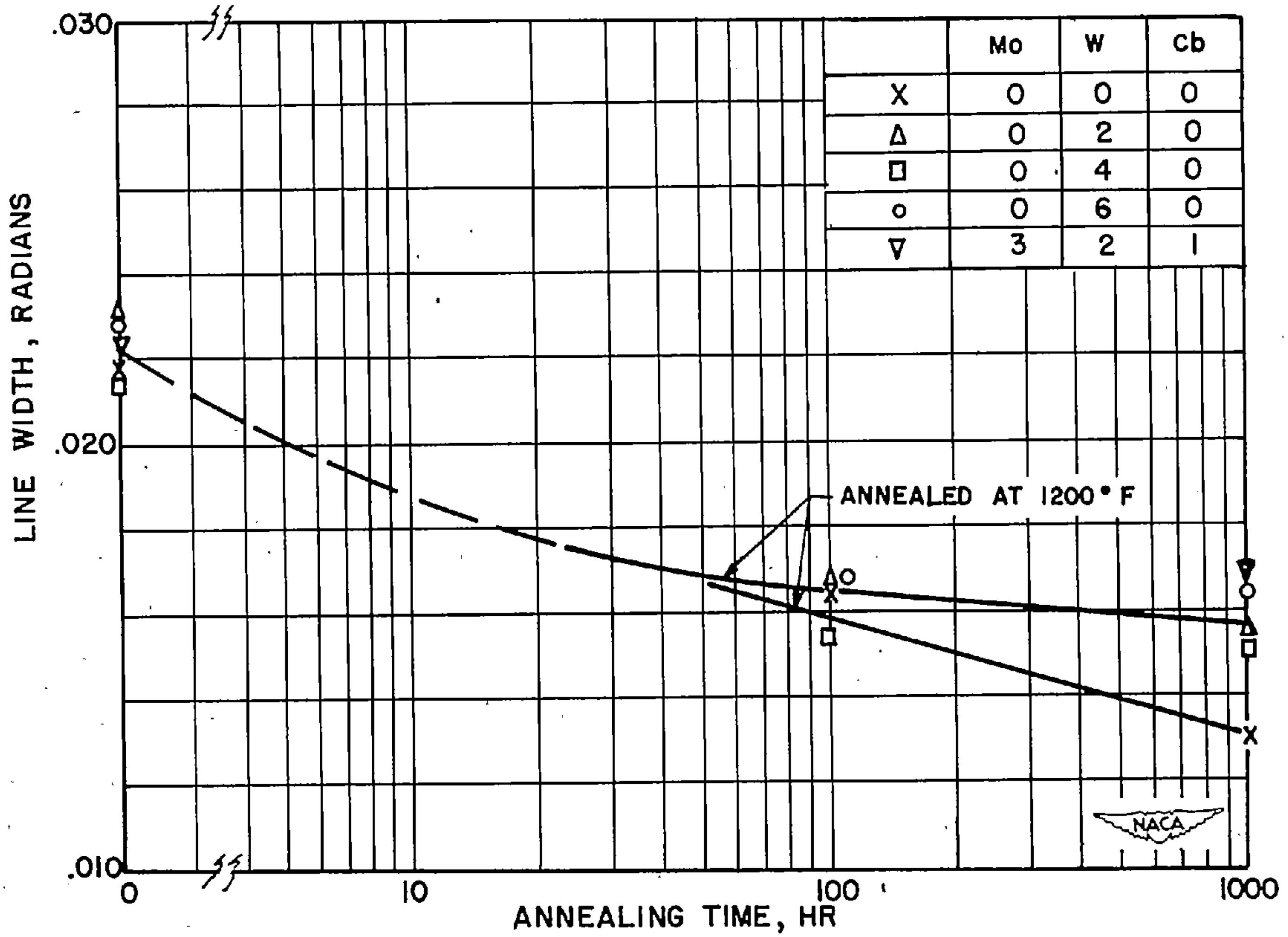

Figure 4.- Effect of annealing on [220] line width of base alloy, modified as indicated, solution-treated 1 hour at 2200° F and reduced 40 percent at 80° F.