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THERMOMECHANICAL PROCESSING OF THE NICKEL-BASE ALLOY U-700

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16. Abstract

Thermomechanical processing (TMP) greatly improved the yield strength and stress-rupture life of the U-700 alloy. At 650° C (1200° F), the best TMP schedule gave about a 3700-hr rupture life at a stress of 827 MN/m^2 (120 ksi). With a conventional heat treatment, the rupture life was about 400 hr under similar test conditions. TMP increased the yield strength of the alloy by at least 50 percent at temperatures of 540° to 760° C (1000° to 1400° F). Correspondingly, the tensile ductility (elongation) of the alloy was reduced from about 35 percent to about 5 to 15 percent.

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SUMMARY

The response of the precipitation-hardened, nickel-base alloy U-700 to thermomechanical processing (TMP) was evaluated. The alloy was cold-worked (20-, 35-, and 50-percent reductions) by extrusion at two different steps in the conventional heat-treating schedule (STA). TMP schedule A consisted of cold-working in the solution-treated condition followed by the carbide-precipitation heat treatment and final aging. In TMP schedule B, the alloy was cold-worked after the carbide-precipitation treatment and then given the final aging treatment. Mechanical properties were compared to the alloy in the conventionally heat-treated condition at ambient temperature and 540°, 650°, and 760° C (1000°, 1200°, and 1400° F).

The best combination of tensile and stress-rupture properties was obtained with 20-percent cold work in the TMP schedules. TMP schedule A samples cold-worked 20 percent gave the best stress-rupture properties. At 650° C (1200° F) and a stress of 827 MN/m^2 (120 ksi) their rupture life was about 3700 hr. Under similar test conditions, the alloy in the STA condition exhibited a life of about 400 hr.

Samples processed according to schedule A or B exhibited similar tensile properties at 540° to 760° C (1000° to 1400° F) for the same levels of cold work. With 20-percent cold work, the yield strength of the TMP sample was about 50 percent greater than that of the alloy in the STA condition. TMP reduced the tensile ductility (elongation) of the alloy from about 35 percent to about 5 to 15 percent.

INTRODUCTION

Special processing techniques that have shown potential for improving the mechanical properties of nickel-base alloys include the powder-metallurgy approach (ref. 1), directional solidification (ref. 2), and thermomechanical processing (refs. 3 to 6). Thermomechanical processing (TMP) consists of the application of combined mechanical working and heat treatment. The purpose of TMP is to provide extensive strain-hardening of the alloy in conjunction with a precipitation-hardening reaction. Precipitation can take place either during or after mechanical working. Both of these effects contribute to the strength of the alloy.

The purpose of this study was to evaluate the response of a high-strength, commercially available nickel-base alloy to TMP. Alloy U-700, a precipitation-hardened alloy with good high-temperature strength, was selected. The alloy was cold-worked at two different steps in the conventional heat-treating schedule. Cold-working was accomplished by hydrostatic extrusion. Mechanical properties at temperatures from 540° to 760° C (1000° to 1400° F) and microstructural examinations were used in evaluating the response of this alloy to TMP.

EXPERIMENTAL PROCEDURE

Material and Conventional Heat Treatment

Alloy U-700 in the form of commercial bar stock (wrought), 1.91 cm (3/4 in.) in diameter, was used in this study. The actual composition of this alloy in weight percent was as follows:

Cobalt	Chromium	Molybdenum	Aluminum	Titanium	Carbon	Zirconium	Boron	Iron	Nickel
15.2	14.8	4.1	4.18	3.34	0.13	0.04	0.015	0.01	Balance

The conventional heat treatment used for alloy U-700 is designated STA for solution treating and aging. It consists of a 1180° C (2150° F) solution treatment for 4 hr, aging at 1065° C (1950° F) for 4 hr to allow for coarse γ' precipitation, aging at 845° C (1550° F) for 24 hr to allow for grain-boundary carbide precipitation, and final aging at 760° C (1400° F) for 16 hr to allow for fine γ' precipitation. Each heat-treating step is followed by air cooling to room temperature.

Thermomechanical Processing

The TMP schedule used in this study is outlined in table I. For reference, the STA schedule is included. As shown, TMP was accomplished by cold-working the alloy at different steps in the STA schedule. It should be emphasized that no attempt was made to determine an optimum TMP schedule for U-700. The schedules used are somewhat arbitrary and represent one approach of applying TMP techniques to alloys of this type.

TMP schedule A consisted of cold-working the alloy in the solution-treated condition. Subsequent aging was done at the lower temperatures, 845° and 760° C (1550° and 1400° F), since the cold-worked alloy would recrystallize at the temperature required for coarse γ' precipitation. Recrystallization would eliminate any strength improvement from strain-hardening. Possibly, an undesirable feature of schedule A is that cold work usually causes intragranular carbide precipitation in this type of alloy upon subsequent heat treatment. Intragranular carbide precipitation might decrease the amount of grain-boundary carbides present. Grain-boundary carbides are important for balanced high-temperature properties (ref. 7).

In TMP schedule B, the alloy was cold-worked after the coarse γ' and grain-boundary carbide precipitation heat treatments. After cold-working, the samples were given the final 760° C (1400° F) aging treatment.

Cold-working levels for both TMP schedules A and B were 20-, 35-, and 50-percent reductions in area. Cold-working was accomplished by the hydrostatic extrusion process described in the next section. Heat treatment after cold-working was done by placing the extrusions in a furnace preheated to 370° C (700° F). After preheating, the samples were heated to the aging temperature, 845° C (1550° F), at the rate of 150° C/hr (300° F/hr). This procedure was used to prevent cracking of the cold-worked samples. Without preheating and the slow heating rate to the aging temperature, the extrusions were prone to axial cracking, particularly those receiving greater amounts of deformation. After aging at 845° C (1550° F), the samples could be placed directly into a 760° C (1400° F) furnace without cracking to complete the heat-treating cycle. All of the heat treatments were done in air.

Hydrostatic Extrusion

Cold-working was accomplished by the hydrostatic extrusion process illustrated in figure 1. As shown, the billet is surrounded by a fluid pressurized by a carbide piston. The piston does not touch the billet. Extrusion of the billet results from the pressure differential across the die. Deformation forces much greater than those found in conventional cold-working processes can be developed by hydrostatic extrusion. For example, fluid pressures can be as high as 2760 MN/m^2 (400 ksi). A more complete description of the hydrostatic extrusion process, equipment, and potential applications is given in references 8 to 10.

Alloy U-700 is difficult to cold-work even in the solution-treated condition. By using the hydrostatic extrusion process, at least a 50-percent reduction in area can be obtained in one step. The U-700 extrusion billets used in this study were 1.84 cm (0.72 in.) in diameter and 10.16 cm (4 in.) long. A 40° included angle was machined on this billet nose to mate with the entrance angle of the extrusion die. The pressurizing fluid was castor oil, and a lead coating (0.001 cm, 0.0005 in., thick) was used for billet lubrication. Extrusion pressures were about 1580 MN/m^2 (230 ksi) for 50-percent cold work, 1100 MN/m^2 (160 ksi) for 35-percent cold work, and 690 MN/m^2 (100 ksi) for 20-percent cold work. For the same level of cold work, there was no appreciable difference in the extrusion pressure required for samples processed according to TMP schedule A or schedule B. Typical extrusions from the 35 billets processed in this study are shown in figure 2.

Evaluation

Mechanical properties of the TMP samples were evaluated by conventional tensile tests at ambient temperature and 540°, 650°, and 760° C (1000°, 1200°, and 1400° F). The tensile specimens conformed to ASTM designation E8. Most of the samples had a 0.40-cm (0.16-in.) gage diameter. Testing was done in an Instron machine at a crosshead speed of 0.13 cm/min (0.05 in./min). Similar type specimens were tested in stress-rupture. Most of the stress-rupture tests were conducted at 650° C (1200° F) and at a stress of 827 MN/m^2 (120 ksi). All the tests were done in air.

The effect of TMP on the microstructure of U-700 was examined by both optical and electron microscopy. However, a quantitative correlation of the mechanical properties to the microstructure and morphology of the precipitates present was beyond the scope of this study.

RESULTS

Mechanical Properties

Tensile and stress-rupture properties for the U-700 alloy in the STA and TMP conditions are summarized in tables II and III and figures 3 to 5.

Tensile properties. - For the same level of cold work there was no appreciable difference in the high-temperature tensile properties (up to 760° C, 1400° F) of the alloy processed by either schedule A or schedule B. However, the amount of cold work had a pronounced effect, as shown in figures 3 and 4. In figure 3, the TMP schedules A and B yield strength data at temperatures from 540° to 760° C (1000° to 1400° F) are shown in two banded regions that represent different levels of cold work. As shown, the higher levels of cold work (35 and 50 percent) for both schedules A and B resulted in yield strengths about twice that of the alloy in the STA condition up to about 650° C (1200° F). The large decrease in strength at 760° C (1400° F) suggests loss of strain-hardening by recovery and possibly some overaging. In comparison, 20-percent cold work gave about a 50-percent increase in yield strength over the entire temperature range up to 760° C (1400° F).

The ultimate tensile strength data for TMP schedules A and B are shown in a similar manner in figure 4. Compared to the alloy in the STA condition, the increase in tensile strength by TMP was not as great as the improvement in yield strength. Cold-working 20 percent (schedule A or schedule B) gave about a 25-percent increase in tensile strength up to 760° C (1400° F). The higher levels of cold work resulted in about a 40-percent strength increase at 540° to 650° C (1000° to 1200° F).

TMP reduced the tensile ductility of the alloy as shown in table II. For example, in the STA condition, the alloy exhibited about 35-percent elongation at 650° C (1200° F). The elongation values for the TMP samples at 650° C ranged from 5 to 15 percent.

Stress-rupture properties. - TMP improved the 650° C (1200° F) stress-rupture life of the alloy as shown in figure 5. At 650° C (1200° F) and a stress of 827 MN/m^2 (120 ksi), the alloy in the STA condition had a life of about 400 hr. TMP samples processed according to schedule A with 20-percent cold work exhibited about a 3700-hr life under similar test conditions. Samples processed according to TMP schedule A gave consistently better stress-rupture lives than those processed by TMP schedule B for all three levels of cold work. Greater amounts of cold work in either schedule reduced the stress-rupture life, but in all cases, the stress-rupture lives of TMP samples at 650° C (1200° F) were greater than that produced by the STA treatment. The ductility, as measured by total elongation in the stress-rupture test, was about 5 percent for the alloy in the STA condition. Similar elongations were exhibited by the TMP samples receiving 20-percent cold work. TMP samples cold-worked 35 or 50 percent had a total elongation ranging from 6 to 20 percent.

Similar tests were conducted at 650° C (1200° F) on samples receiving the schedule A heat treatment only for comparison to the TMP schedule A samples. As shown in table III, the schedule A heat-treated samples exhibited stress-rupture lives (about 350 hr) less than the alloy in the STA or TMP schedule A conditions.

Limited stress-rupture tests were conducted at 760° C (1400° F) and a stress of 552 MN/m^2 (80 ksi). Under these conditions, the U-700 alloy in the STA condition is reported to have a life of about 100 hours (ref. 11). The four TMP schedule B samples with 35- and 50-percent cold work did not exceed 70 hours in the stress-rupture tests (table III). Only one TMP schedule A sample with 20-percent cold work was tested at

760° C (1400° F). But the stress-rupture life of this sample was 190 hr, nearly double the reported value for U-700 in the STA condition.

Metallographic Evaluation

The microstructure of U-700 in the STA condition and after TMP (for 35-percent cold work) is shown in figure 6. In the STA condition, the structure consists of a duplex grain size with coarse γ' precipitation within the grains. TMP schedule A resulted in elongation of the grains in the direction of working and heavy deformation bands within the grains. The density of the deformation bands increased with increasing amounts of cold work. Coarse γ' was not observed in the structure. After processing according to TMP schedule B, the grains are elongated in the direction of cold work with no evidence of deformation bands.

Electron photomicrographs of the STA and TMP structures (35-percent cold work) are shown in figure 7. In the STA condition, the structure exhibits discontinuous carbide precipitation at the grain boundaries and the cube-shaped, coarse γ' within the grains. After TMP according to schedule A, the structure shows discontinuous carbide precipitation both along the grain boundaries and within the grains, but no evidence of coarse γ'. However, a fine, unresolved γ' phase is probably present in the structure. The intragranular carbides precipitated along the deformation bands. A finer carbide precipitation along the deformation bands was associated with smaller amounts of cold work in the TMP schedule A samples. At all levels of cold work, the TMP schedule B structure was very similar to the structure observed in the STA condition.

As shown in figure 8, the structure of the TMP samples appears unchanged after the long time stress-rupture tests at 650° C (1200° F).

DISCUSSION

Significant improvements in the yield strength and stress-rupture life of the U-700 alloy were obtained by TMP. TMP schedule A with 20-percent cold work gave the best combination of tensile and stress-rupture properties. The yield strength was about 50 percent greater than the alloy in the STA condition at temperatures from 540° to 760° C (1000° to 1400° F) (fig. 3). At 650° C (1200° F), the stress-rupture life showed a ninefold improvement over the alloy in the STA condition. Both the tensile data and the stress-rupture test at 760° C (1400° F) suggest stability of the structure developed by TMP schedule A (20-percent cold work) to 760° C (1400° F).

Probably, the greatly improved stress-rupture life developed by TMP schedule A is related to the fine intragranular carbide precipitation in conjunction with strain-hardening. In addition, the presence of fine γ' in the strain-hardened matrix may contribute to the strength increases noted. TMP schedule B, which exhibited no intragranular carbide precipitation and probably had a lesser amount of fine γ', gave a moderate improvement in stress-rupture life. Although the greater amounts of cold-working (35 and 50 percent) in the U-700 TMP schedules gave higher tensile properties, the stress-rupture life was not as good as that developed by 20-percent cold work. Probably, the additional strain energy from greater amounts of cold work caused "overaging" effects such as the apparent growth of larger intragranular carbides in TMP schedule A samples subject to 35- and 50-percent cold work. A more detailed study is required to correlate the structure developed by TMP to determine the relation of the TMP schedule to structural stability.

Hotzler, Maciag, Fischer, and Troc (ref. 6) obtained improved tensile properties in thermomechanically processed Astroloy, an alloy similar in composition to U-700. In their TMP schedule, the alloy was warm-worked at 815° C (1500° F). The tensile and yield properties reported for Astroloy in the TMP condition are comparable to those of the TMP U-700 reported herein. Also, the best results for the TMP Astroloy were obtained with 20-percent work in the TMP schedule. However, the stress-rupture life of the TMP Astroloy at 815° C (1500° F) was only one-fourth that of the alloy in the STA condition.

A direct comparison of the TMP schedule for U-700 and Astroloy cannot be made because of the differences in heat treatment and the temperatures of working and stress-rupture testing. But one of the microstructural differences was the absence of intragranular carbide precipitation in Astroloy subjected to TMP. Intragranular precipitation of carbides along the deformation bands produced by cold-working was characteristic of the U-700 alloy subjected to TMP schedule A. Even without the presence of intragranular carbides, TMP schedule B (20-percent cold work) gave a threefold improvement in stress-rupture life for the U-700 alloy. Thus, the deformation temperature may play a major role in the stability of the structure developed by TMP alloys of this type since the U-700 alloy was cold-worked and Astroloy was warm-worked at 815° C (1500° F).

The relatively small amounts of cold work required to obtain the best properties in the TMP U-700 (20-percent reduction) is significant. More conventional deformation processes such as rolling might be used in the TMP schedule rather than the hydrostatic extrusion process that was used in this study to evaluate a wider range of cold-working levels.

Although TMP schedule A gave improved properties, this may not be the optimum TMP for the U-700 alloy. A more optimum schedule might give a better balance in strength and ductility. In addition to development of a more optimum processing schedule, future work should be directed toward defining the relation of structure to the properties developed by TMP techniques.

SUMMARY OF RESULTS

The response of the precipitation-hardened, nickel-base alloy U-700 to thermomechanical processing (TMP) was evaluated. The alloy was cold-worked (20-, 35-, and 50-percent reductions) by extrusion at two different steps in the conventional heat-treating schedule (STA). TMP schedule A consisted of cold-working in the solution-treated condition followed by the carbide-precipitation heat treatment and final age. In TMP schedule B, the alloy was cold-worked after the carbide precipitation treatment and then given the final aging treatment. Mechanical properties were compared to the alloy in the conventionally heat-treated condition.

The following results were obtained:

1. The best combination of tensile and stress-rupture properties was obtained with 20-percent cold work in the TMP schedules.

2. The stress-rupture lives at 650° C (1200° F) of samples processed according to TMP schedule A were consistently better than those obtained by TMP schedule B.

3. TMP schedule A samples cold-worked 20 percent gave the best stress-rupture properties. At 650° C (1200° F) and a stress of 827 MN/m^2 (120 ksi), their rupture life showed a ninefold improvement over the alloy in the STA condition.

4. With 20-percent cold work, the yield strength of the TMP samples was about 50 percent greater than the alloy in the STA condition from 540° to 760° C (1000° to 1400° F).

5. For the same level of cold work there was no appreciable difference in the tensile properties at 540° to 760° C (1000° to 1400° F) of samples processed according to schedule A or B.

6. In the STA condition, the alloy exhibited about 35-percent elongation in the tensile tests. The TMP samples exhibited about 5- to 15-percent elongation.

Lewis Research Center,
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Cleveland, Ohio, June 15, 1971,
129-03.

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TABLE I. - THERMOMECHANICAL PROCESSING (TMP) SCHEDULE FOR U-700

[All samples solution-treated at 1175° C (2150° F) for 4 hr in air prior to processing.]

Temperature, °C (°F)	Time, hr	STA	TMP schedule A	TMP schedule B
1065 (1950)	4	γ' precipitation	--------------------	γ' precipitation
Ambient	--	-------------------	Cold work (0-, 20-, 35-, and 50-percent reduction in area)	-------------------
845 (1550)	24	Carbide precipitation	Carbide precipitation	Carbide precipitation
Ambient	--	-------------------	-------------------	Cold work (20-, 35-, and 50-percent reduction in area)
760 (1400)	16	Final aging	Final aging	Final aging

TABLE II. - TENSILE PROPERTIES OF CONVENTIONALLY HEAT-TREATED AND THERMOMECHANICALLY PROCESSED U-700 ALLOY

Sample condition	Percent cold work	Temperature		0.2 percent yield strength		Tensile strength		Elongation, percent	Reduction in area, percent
		°C	°F	MN m^2	ksi	MN m^2	ksi		
STA	0	26	80	930	134.6	1440	208.9	20.0	21.8
				945	136.8	1420	206.0	20.0	18.9
		540	1000	935	135.4	1335	193.5	28.0	18.1
		650	1200	895	130.5	1250	181.6	34.0	30.8
		760	1400	720	104.0	800	116.3	25.0	26.7
				910	132.5	1095	158.7	25.0	25.9
TMP schedule A	20	26	80	1340	195.0	1640	238.0	12.0	14.4
		540	1000	1210	175.0	1540	223.5	8.0	7.3
		650	1200	1220	177.5	1495	217.0	13.3	14.2
				1240	180.0	1520	220.0	10.7	11.8
		760	1400	1055	153.0	1130	163.5	20.0	28.5
TMP schedule A	35	540	1000	1450	210.0	1635	237.6	3.0	4.1
				1450	210.0	1680	244.0	7.8	2.5
		650	1200	1390	202.0	1540	223.0	7.8	6.6
				1410	204.0	1550	225.0	6.0	16.2
		760	1400	730	106.0	950	137.7	8.0	7.0
				885	128.6	1020	147.5	10.0	25.4
TMP schedule A	50	540	1000	1670	242.0	1740	252.0	1.5	2.4
		650	1200	1330	193.0	1480	215.4	14.0	32.0
				1510	219.0	1600	232.0	12.5	16.9
		760	1400	920	133.8	1060	154.4	23.0	6.5
TMP schedule B	20	26	80	1430	207.5	1615	234.0	13.3	14.4
		540	1000	1310	190.0	1520	220.0	8.0	4.9
		650	1200	1290	187.5	1460	211.0	13.3	17.9
				1300	188.5	1460	211.0	16.0	22.1
		760	1400	1070	155.0	1130	164.0	17.3	24.8
TMP schedule B	35	26	80	1740	252.0	1790	260.0	4.0	7.4
		540	1000	1500	225.0	1660	240.8	8.0	3.9
		650	1200	1390	202.0	1510	219.0	14.7	18.6
		760	1400	835	121.0	1040	151.2	16.0	21.3
TMP schedule B	50	540	1000	1760	255.0	1815	263.6	4.0	3.5
		650	1200	1635	237.0	1720	249.0	8.0	4.7
		760	1400	835	121.0	886	128.7	33.3	27.7

TABLE II. - STRESS-RUPTURE PROPERTIES OF CONVENTIONALLY HEAT-TREATED AND THERMOMECHANICALLY PROCESSED U-700 ALLOY

Sample condition	Percent cold work	Temperature		Stress		Life, hr	Elongation, percent
		°C	°F	MN/m^2	ksi		
STA	0	650	1200	827	120	462	6.0
						396	5.0
TMP schedule A	0	650	1200	827	120	364	14.1
						338	18.8
TMP schedule A	20	650	1200	827	120	3786	4.0
		650	1200	827	120	3698	5.3
		760	1400	552	80	190	4.7
TMP schedule A	35	650	1200	827	120	1290	13.3
						1192	6.7
TMP schedule A	50	650	1200	827	120	673	9.4
						1004	18.7
TMP schedule B	20	650	1200	827	120	1425	4.0
						48	20.0
						1372	4.0
TMP schedule B	35	650	1200	827	120	905	6.7
		650	1200	827	120	1051	8.0
		760	1400	552	80	7	----
		760	1400	552	80	>3, <67	6.7
TMP schedule B	50	650	1200	827	120	764	12.0
		650	1200	827	180	650	17.4
		760	1400	552	80	24	12.0
		760	1400	552	80	>27, <43	10.7

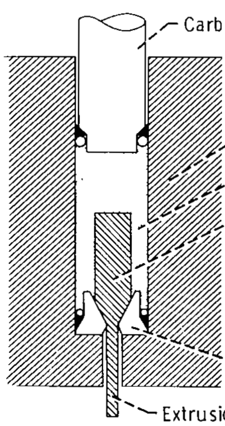

Figure 1. - Schematic diagram of hydrostatic extrusion.

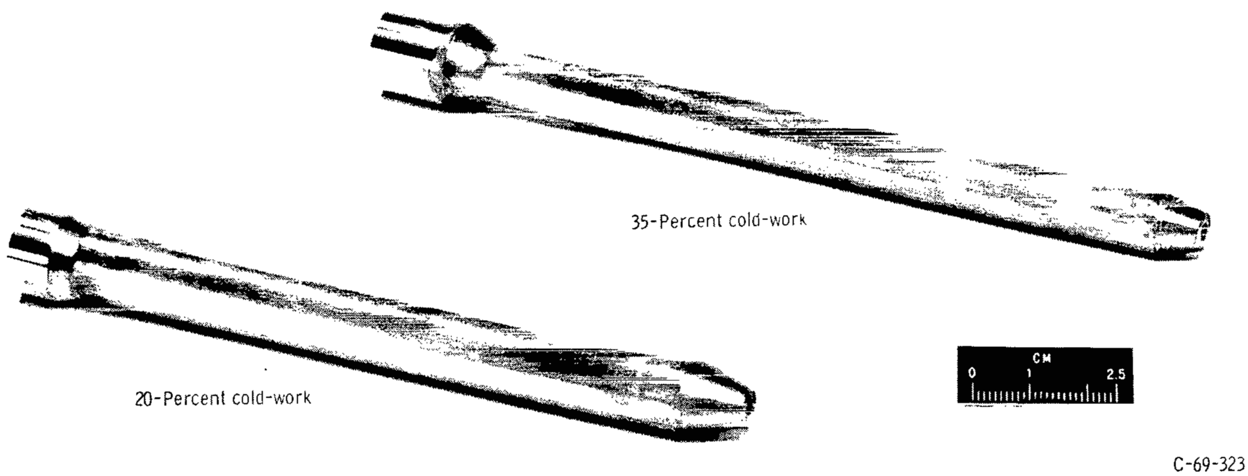

Figure 2. - Typical U-700 extrusions produced by hydrostatic extrusion process.

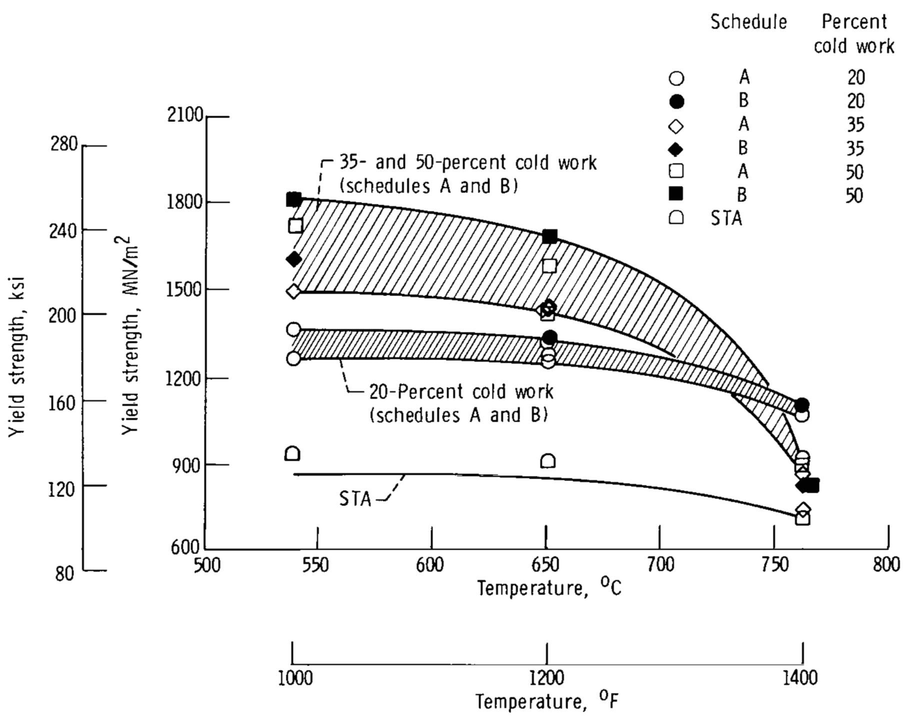

Figure 3. - Effect of thermomechanical processing on yield strength of alloy U-700.

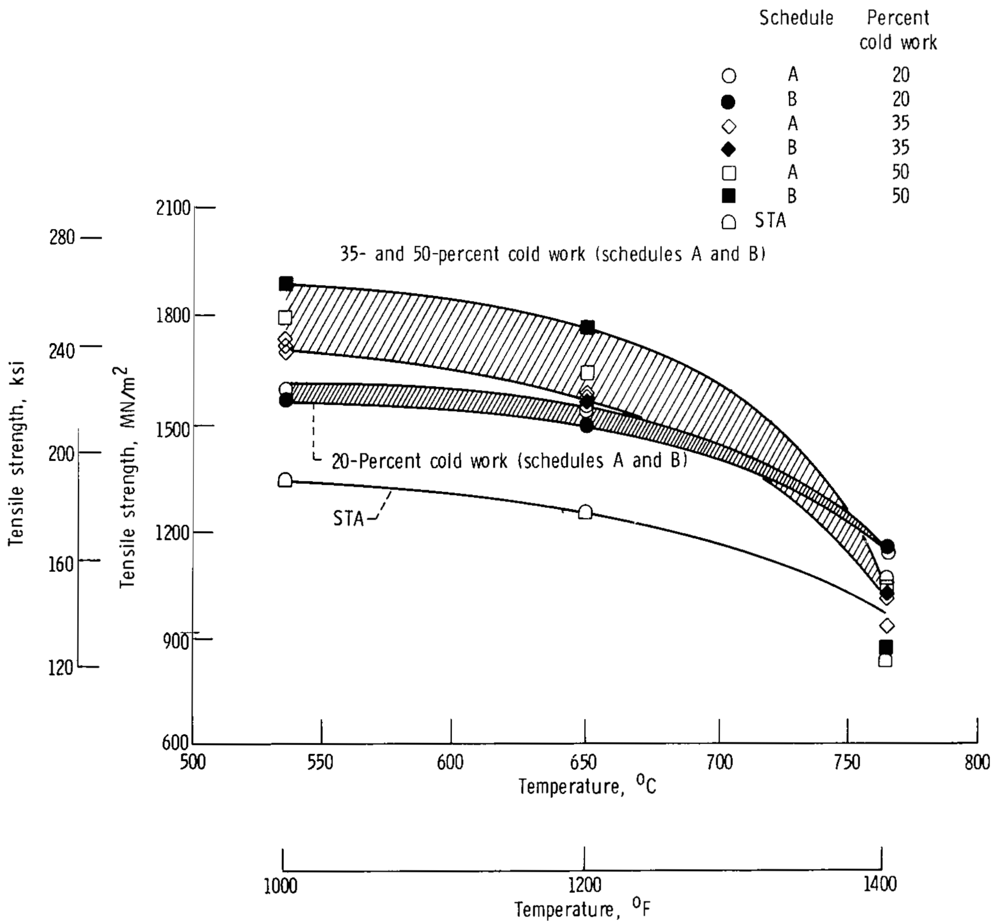

Figure 4. - Effect of thermomechanical processing on tensile strength of alloy U-700.

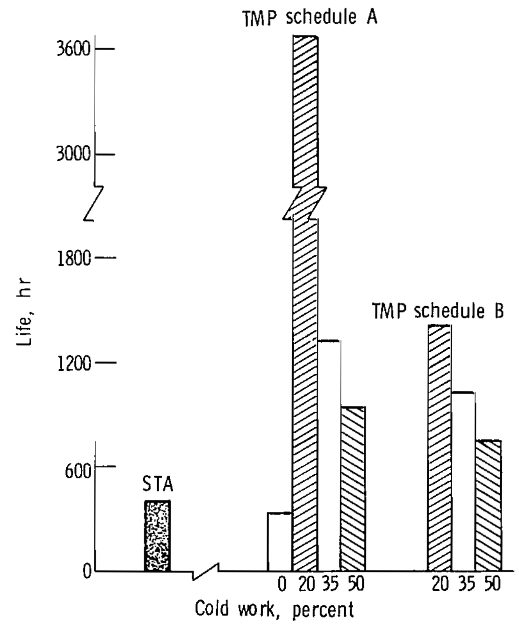

Figure 5. - Effect of thermomechanical processing on stress-rupture life of alloy U-700. Temperature, 650° C (1200° F); stress, 827 MN/m^2 (120 ksi).

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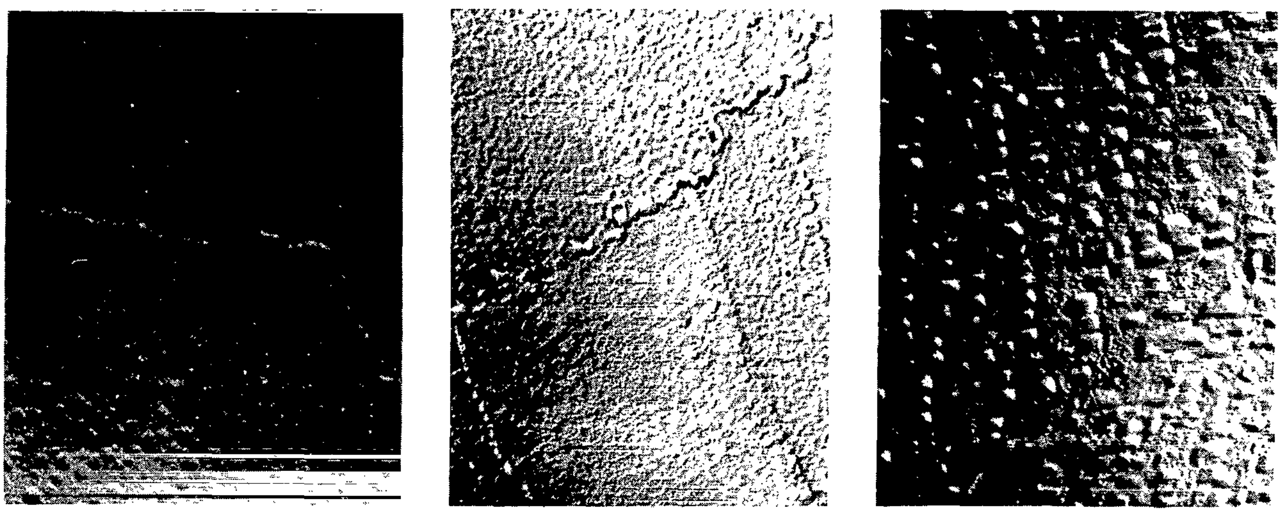
TMP schedule A (35-percent cold-worked)

TMP schedule B (35-percent cold-worked)

Figure 6. - Effect of thermomechanical processing on microstructure of alloy U-700. Electrolytically etched; etchant, 36 percent water; 36 percent glycerine; 10 percent nitric acid; 10 percent hydrofluoric acid; X250.



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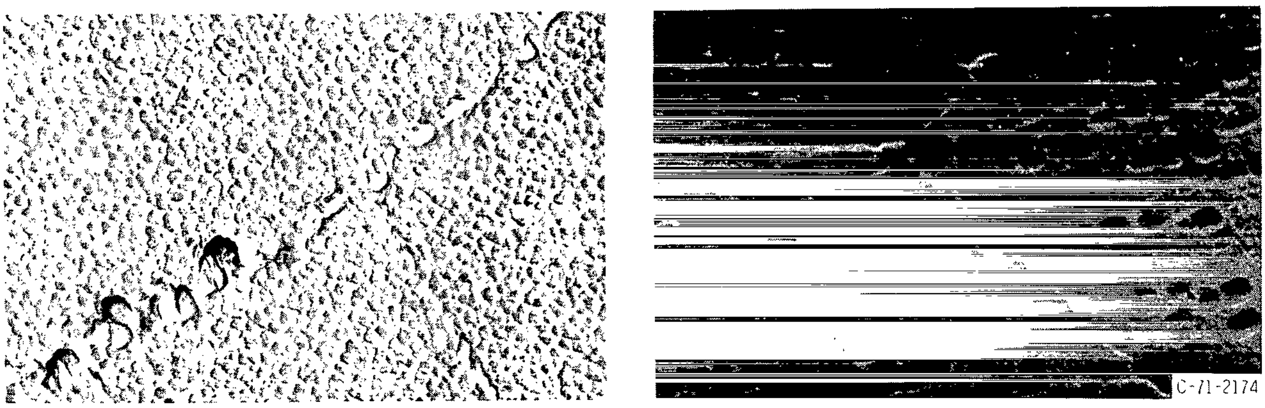
STA

Schedule A (35-percent cold-worked)

Schedule B (35-percent cold-worked)

Figure 7. - Effect of thermomechanical processing on microstructure of alloy U-700. Two-stage carbon replica; X10 000.



(a) TMP schedule A; 20-percent cold-worked; 3800 hours.

(b) TMP schedule B; 20-percent cold-worked; 1425 hours.

Figure 8. - Effect of exposure in stress-rupture tests at 650° C (1200° F) and 827 MN/m^2 (120 ksi) on microstructure of alloy U-700 after thermo-mechanical processing. Two-stage carbon replica; X10 000.