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CHARACTERIZATION OF THE ANELASTIC
AND THERMOELASTIC PROPERTIES
OF A URANIUM-NIOBIUM-
ZIRCONIUM ALLOY

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ABSTRACT

Anelastic and thermoelastic properties of the uranium-7.5 niobium-2.5 zirconium alloy have been measured by four types of tests: (1) effect of plastic strain on the shape of the stress-strain curves as a function of the permanent strain for two levels of hydrogen content, (2) a study of the hysteresis loop as a function of the number of stress cycles, (3) measurement of the springback of plastically deformed specimens as a function of time, and (4) measurement of the springback of elastically deformed specimens as a function of time. Possible mechanisms have been proposed for the results of these tests, and a set of critical experiments has been suggested to elucidate this behavior.

CONTENTS

SUMMARY	4
INTRODUCTION	5
ANELASTIC AND THERMOELASTIC PROPERTIES OF A URANIUM ALLOY	6
Experimental Procedure	6
Results	6
Two-Percent-Strain-Increment Tensile Studies	6
Tension Studies	9
U-Bend (180-Degree) Studies	14
Elastic Bending Studies	15
Discussion of Results	15
Tensile Studies	15
U-Bend (180-Degree) Studies	21
Elastic Bending Studies	24
REFERENCES	26

SUMMARY

Anelastic and thermoelastic properties of the uranium-7.5 niobium-2.5 zirconium alloy (mulberry) have been measured by four types of tests. These tests brought out the following observations:

1. The effect of plastic strain on the shape of the stress-strain curves was evaluated as a function of the permanent strain for two levels of hydrogen content. Three different elastic moduli were observed on each curve after the initial permanent set. About 2% strain can be recovered elastically.
2. During repeated stressing to 690 MPa, the area of the hysteresis loop decreases exponentially with the number of stress cycles.
3. The springback of plastically deformed specimens (U bends) decreases linearly with the aging time if they are restrained during aging. The springback actually becomes negative so that the specimen continues to close up during long-time aging.
4. Elastically deformed specimens also show a decrease in springback with the aging time. However, the kinetics of this reaction is much faster than for the plastic recovery.

Possible mechanisms have been proposed for the results observed from these tests, and a set of critical experiments has been suggested to elucidate this behavior.

INTRODUCTION

Much effort has been devoted toward developing a stainless uranium alloy suitable for use as an engineering material. Unalloyed uranium has rather poor mechanical properties and corrodes readily when exposed to the atmosphere. A desirable solution is to alloy the uranium with such elements as molybdenum, niobium, and zirconium which stabilizes the high-temperature gamma phase at room temperature. Such binary alloys possess improved strength, ductility, and corrosion resistance. In addition, their physical properties can be altered by heat treatment.

Investigations into ternary systems involving uranium with molybdenum, niobium, and zirconium at 10 wt % maximum alloy addition indicate that the uranium-7.5 niobium-2.5 zirconium alloy (U-7.5 Nb-2.5 Zr) best meets the criteria for a multipurpose "stainless" uranium alloy. However, this alloy is metastable gamma phase (bcc) on quenching, and low-temperature heat treatment causes this structure to decompose to several products. The crystallography of these phases has been determined by Yakel.⁽¹⁾ A time-temperature transformation diagram was presented by Dean.⁽²⁾

Dimensional instability is a problem common to many metastable alloys. This problem stems from the propensity for the metastable phase to transform to the equilibrium phase, and may be complicated by the tendency for intermediate metastable phases to form.

The purpose of this investigation, performed at the Oak Ridge Y-12 Plant^(a) was to: (1) characterize the anomalous stress-strain behavior of this alloy, including abrupt changes in the Young's modulus at stresses far below the flow stress, and (2) characterize the effects of stresses, both applied and residual, on the manner in which the metastable phases transform during heat treatment.

Uranium-niobium,^(3,4) uranium-zirconium,^(3,4) and uranium-niobium-zirconium⁽⁵⁾ phase diagrams have been determined. The physical metallurgy and mechanical properties of uranium-niobium binary alloys have been studied,^(6,7) as well as their thermoelastic properties.⁽⁸⁾ The physical properties of the U-7.5 Nb-2.5 Zr alloy have also been reported.^(9,10)

(a) Operated by the Union Carbide Corporation's Nuclear Division for the US Energy Research and Development Administration.

ANELASTIC AND THERMOELASTIC PROPERTIES OF A URANIUM ALLOY

EXPERIMENTAL PROCEDURE

All material used in these experiments was made from uranium, niobium, and zirconium strips fabricated into electrodes and consumably arc melted into cylindrical electrodes. These electrodes were again arc melted and skull cast in a graphite vertical slab mold. A typical analysis of the starting material is given in Table 1. The alloy was vacuum homogenized for 18 hours at 900° C, cross rolled to a thickness of 13 mm at 850° C, annealed in argon at 800° C for one hour, and water quenched. This treatment produced a grain size of approximately 40 micrometers.

Table 1
TYPICAL URANIUM-7.5 NIO-
BIUM-2.5 ZIRCONIUM SPECI-
MEN ANALYSIS

Element	Concentration ⁽¹⁾
Aluminum	4
Carbon	80
Chromium	< 10
Copper	4
Hydrogen	2
Iron	20
Magnesium	< 2
Molybdenum	< 10
Nickel	12
Nitrogen	11
Oxygen	56
Silicon	50
Tungsten	< 60
Uranium	89.69 wt %
Niobium	7.56 wt %
Zirconium	2.38 wt %

(1) All values in ppm except as noted.

Three types of specimens were used in this study: tensile specimens, which were 6.4 mm in circular cross section and 25-mm-gage-length designs with threaded grips; bend specimens, for the plastically deformed U-bend studies which were strips measuring 25 by 150 by 2.5 mm, and bend specimens for the elastic bends which were strips measuring 25 by 220 by 3.1 mm.

A Tinius-Olsen Electromatic testing machine was used for the room-temperature tensile tests at crosshead speeds of less than 2.5 mm per minute; tensile tests at crosshead speeds greater than 2.5 mm per minute or at elevated temperatures were performed on an MTS hydraulic machine equipped with an infrared furnace.

The 180-degree U-bend specimens were given three-point bends, using the tools shown in Figure 1. The 13-mm bend radius achieved with these tools corresponds to an approximately 10% maximum outer fiber strain.

The elastically deformed specimens were bent using a four-point jig seen in Figure 2 and depicted schematically in Figure 3. A maximum outer-fiber stress of approximately 410 MPa could be applied using these fixtures.

RESULTS

Two-Percent-Strain-Increment Tensile Studies

Tensile specimens were strained in 2% increments, relaxing the load after each cycle, as indicated in Figures 4 and 5. In addition to the initial 800° C argon anneal, half the specimens were reannealed. These anneals were for one hour at 800° C in vacuum plus a quench, or for 10 hours at 800° C in a carbonate salt bath plus a water quench. The purpose of the salt-bath treatment was to charge the specimens with hydrogen. Typical analyses indicated that the salt-bath-annealed material contained 80 to 100 ppm hydrogen, 20 to 40 ppm oxygen, and 2 to 5 ppm nitrogen; gas analyses for the vacuum-annealed material

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Figure 1. RAM, DIE, AND SEAT ASSEMBLY USED TO BEND STRIPS 180 DEGREES TO A 12.7-mm RADIUS.

showed 1 to 3 ppm hydrogen, 20 to 60 ppm oxygen, and 5 to 12 ppm nitrogen. All tests were at room temperature using crosshead speeds that varied from 0.13 to 13 mm/min.

Specimens were tested in the following ten conditions:

1. Vacuum annealed at 800° C for one hour and water quenched.
2. Vacuum annealed at 800° C for one hour, water quenched, then aged for 15 minutes at 200° C.
3. Vacuum annealed at 800° C for one hour, water quenched, then aged for 30 minutes at 200° C.
4. Vacuum annealed at 800° C for one hour, water quenched, then aged for 45 minutes at 200° C.
5. Vacuum annealed at 800° C for one hour, water quenched, then aged for 60 minutes at 200° C.
6. Salt-bath annealed at 800° C for 10 hours and water quenched.
7. Salt-bath annealed at 800° C for 10 hours, water quenched, then aged for one hour at 150° C.

E730

Figure 2. FOUR-POINT ELASTIC BENDING FIXTURE AND SPECIMENS. (1/2X)

Figure 3. DIMENSIONS OF THE FOUR-POINT BENDING APPARATUS. (Specimen Deflection, h)

8. Salt-bath annealed at 800° C for 10 hours, water quenched, prestrained 6%, then aged for one hour at 150° C.
9. Salt-bath annealed at 800° C for 10 hours, water quenched, prestrained 6%, then aged for one hour at 200° C.
10. Salt-bath annealed at 800° C for 10 hours, water quenched, prestrained 6%, then aged for one hour at 250° C.

Figure 4. LOAD CYCLING FOR MULBERRY THAT WAS SALT-BATH ANNEALED FOR TEN HOURS, WATER QUENCHED, THEN AGED FOR ONE HOUR AT 150° C.

Figure 5. LOAD CYCLING FOR MULBERRY THAT WAS VACUUM ANNEALED FOR ONE HOUR AT 800° C, WATER QUENCHED, THEN AGED FOR 45 MINUTES AT 200° C. (Typical Elastic and Anelastic Recovery during Unloading is also Shown)

Results of these tests are listed in Tables 2 and 3. These results are average values for at least two specimens per condition. The methods used for extracting data are illustrated in Figure 6. The lower, middle, and upper moduli were derived using the classical line-tangent method with the positions of the "knees" determined by the intersection of the tangents. The "average modulus" was extracted by inserting a line between the initial loading point and the actual yield point for each loading cycle. The area within the hysteresis loop, which is a measure of anelastic energy expended by the material during a loading and unloading cycle, was determined with a polar-compensating planimeter. Each square inch of area enclosed within the hysteresis loop on the load-extension plot corresponds to 1.7 GJ/m^3 of anelastic energy.

Tension Studies

The strain studies suggested that measurements of the decrease in anelastic energy as a function of the number of strain cycles would be pertinent. For this study, four specimen conditions were studied:

1. Argon annealed at 800°C for one hour, water quenched, then prestrained 2.5%.
2. Argon annealed at 800°C for one hour, water quenched, then prestrained 5.0%.

Figure 6. TYPICAL LOADING CYCLE, SHOWING METHODS USED TO EXTRACT THE MODULI, THE KNEE POSITIONS, AND THE AVERAGE MODULUS.

Table 2
TENSILE-TEST DATA FOR URANIUM-7.5 NIOBIUM-2.5 ZIRCONIUM ALLOY IN VARIOUS HEAT-TREATED AND PRESTRAINED CONDITIONS
(Values are Averaged for at Least Two Specimens)

Test	Specimen									
	1	2	3	4	5	6	7	8	9	10
Annealing Machine	Vacuum	Vacuum	Vacuum	Vacuum	Vacuum	Salt Bath	Salt Bath	Salt Bath	Salt Bath	Salt Bath
Annealing Time (hr)	1	1	1	1	1	10	10	10	10	10
Aging Temperature ($^{\circ}$ C)	-	200	200	200	200	-	150	-	200	250
Aging Time (min)	0	15	30	45	60	0	60	0	60	60
Prestrain (%)	0	0	0	0	0	0	0	6	6	6
Initial Loading Cycle (2% total strain)										
Hardness (R_A)	62.4	63.6	65.4	65.6	65.4	59.9	61.3	64.3	65.8	66.4
Yield Strength at One Percent Strain (MPa) ⁽¹⁾	558	462	462	462	524	524	527	431	458	458
Yield Strength at Two Percent Strain (MPa) ⁽²⁾	754	768	772	806	854	665	796	730	744	748
Lower Modulus (10^6 GPa)	76	56	57	49	66	69	65	54	56	60
Stress where First Knee Occurs (MPa)	-	403	396	589	544	-	-	389	389	282
Middle Modulus (10^6 GPa)	-	28	29	28	27	-	-	30	28	26
Stress where Second Knee Occurs (MPa)	-	-	-	-	-	-	-	-	-	-
Upper Modulus (10^6 GPa)	-	-	-	-	-	-	-	-	-	-
Average Young's Modulus (10^6 GPa) ⁽³⁾	38	39	40	41	43	36	41	36	38	38

(1) The stress at 1% total elastic, anelastic, and plastic strain is a convenient value for materials displaying nonlinear elastic behavior.

(2) In general, this alloy in the annealed condition requires approximately 2% total strain to produce any permanent deformation.

(3) The slope of the stress-strain curve from zero stress to the actual yield stress.

Table 2 (Continued)

Test	Specimen									
	1	2	3	4	5	6	7	8	9	10
<u>Second Loading Cycle (4% total strain)</u>										
Lower Modulus (GPa)	75	[Identical to Initial Loading Cycle]				63	61	56	55	50
Stress where First Knee Occurs (MPa)	341					289	-	279	400	276
Middle Modulus (GPa)	30					27	-	32	31	34
Stress where Second Knee Occurs (MPa)	-					-	-	-	661	668
Upper Modulus (GPa)	-					-	-	-	39	40
Average Modulus (GPa)	38					37	41	37	39	40
Stress at Four Percent Total Strain (MPa)	837	965	1030	1058	1078	813	885	706	875	920
<u>Third Loading Cycle (6% total strain)</u>										
Lower Modulus (GPa)	58	48	51	48	58	53	58	50	49	53
Stress where First Knee Occurs (MPa)	262	334	351	462	472	262	365	276	289	231
Middle Modulus (GPa)	30	28	28	26	26	28	25	34	34	35
Stress where Second Knee Occurs (MPa)	672	706	765	858	858	-	703	706	672	744
Upper Modulus (GPa)	35	36	36	31	30	-	30	36	36	39
Average Modulus (GPa)	37	36	36	34	35	35	34	39	39	39
Stress at Six Percent Total Strain (MPa)	854	958	1020	1047	1061	796	844	851	882	930

Table 2 (Continued)

Test	Specimen									
	1	2	3	4	5	6	7	8	9	10
Fourth Loading Cycle (8% total strain)										
Lower Modulus (GPa)	52	46	48	45	52	48	53	46	45	50
Stress where First Knee Occurs (MPa)	262	296	282	382	351	262	307	282	282	165
Middle Modulus (GPa)	34	30	32	30	31	31	28	34	34	37
Stress where Second Knee Occurs (MPa)	699	706	754	789	830	689	686	682	665	741
Upper Modulus (GPa)	36	39	40	36	34	36	34	38	37	39
Average Modulus (GPa)	38	36	38	35	36	36	36	38	39	39
Stress at Eight Percent Strain (MPa)	861	944	1006	1037	1047	813	896	844	889	937
Fifth Loading Cycle (8% total strain)										
Lower Modulus (GPa)	48	43	44	43	54	48	49	44	45	45
Stress where First Knee Occurs (MPa)	241	300	310	376	320	238	276	248	196	193
Middle Modulus (GPa)	34	32	34	34	34	31	30	34	36	38
Stress where Second Knee Occurs (MPa)	-	665	730	686	765	630	624	-	-	-
Upper Modulus (GPa)	-	37	41	38	42	38	33	-	-	-
Average Modulus (GPa)	39	36	39	37	37	37	35	37	39	40
Ultimate Tensile Strength (MPa)	861	965	1034	1058	1071	813	896	861	889	937

3. Salt-bath annealed at 800° C for ten hours, water quenched, then prestrained 2.5%.

4. Salt-bath annealed at 800° C for ten hours, water quenched, then prestrained 5.0%.

The specimens were prestrained because the earlier experimental data showed that changes in the elastic behavior of this alloy do not occur until after the specimen has been strained plastically. Two prestrain values are listed, 2.5 and 5.0%. On unloading, the 2.5% prestrained specimens recovered approximately 1.5% of their strain and the 5.0% prestrained specimens recovered approximately 1.8% of their initial strain. Consequently, the specimens began the cycling with a 1.0 and 3.2% permanent set.

Table 3
ANELASTICITY DATA FOR URANIUM-7.5 NIOBIUM-2.5 ZIRCONIUM ALLOY IN VARIOUS
HEAT-TREATED AND PRESTRAINED CONDITIONS

Test	Specimen									
	1	2	3	4	5	6	7	8	9	10
<u>Initial Loading Cycle (2% total strain)</u>										
Total Elastic and Anelastic Recovery upon Unloading (%)	1.8	2.0	2.0	2.0	2.2 ⁽¹⁾	1.8	1.9	1.8	1.8	1.8
Anelastic Energy Represented in Hysteresis Loop ⁽²⁾ (MJ/m ³)	1203	1108	1239	929	1549	1590	965	881	1167	973
<u>Second Loading Cycle (4% total strain)</u>										
Total Elastic and Anelastic Recovery upon Unloading (%)	2.2	2.7	2.9	3.0	3.0	2.2	2.5	2.2	2.3	2.3
Anelastic Energy Represented in Hysteresis Loop (MJ/m ³)	1690	2687	3541	3919	4599	1966	2697	1171	1307	1509
<u>Third Loading Cycle (6% total strain)</u>										
Total Elastic and Anelastic Recovery upon Unloading (%)	2.2	2.6	2.6	2.8	2.8	2.2	2.5	2.2	2.3	2.3
Anelastic Energy Represented in Hysteresis Loop (MJ/m ³)	1380	2303	2452	3296	3597	1777	2322	1062	1190	1372
<u>Fourth Loading Cycle (8% total strain)</u>										
Total Elastic and Anelastic Recovery upon Unloading (%)	2.2	2.6	2.6	2.8	2.8	2.2	2.5	2.2	2.3	2.3
Anelastic Energy Represented in Hysteresis Loop (MJ/m ³)	1333	2018	2172	2461	3655	1490	2112	1095	1056	1144

(1) Specimen was initially strained to 2.2% instead of 2.0%. All deformation was recovered.

(2) Hysteresis loop delineated by unloading plot from one cycle and the loading plot from the next cycle.

After each specimen had been prestrained it was relaxed and then cycled from 0 to 690 MPa and back to 0 MPa stress at 0.1 Hz for at least 2000 cycles. The MTS machine, programmed with a triangular waveform, was used for this experiment. The load-versus-strain behavior was oscillographically recorded. Each square centimeter on the oscillograph represented 207 MJ/m³ of anelastic energy.

Selected oscillographic frames as a function of the number of cycles are presented in Figure 7. Results of this investigation are given in Table 4 and plotted in Figures 8 and 9. The areas within the hysteresis loops were measured planimetrically. Error analysis indicated that areas measured by this method are accurate to approximately $\pm 20\%$.

U-Bend (180-Degree) Studies

Strips described earlier were bent 180 degrees to a radius of 13 mm (corresponding to a 10% maximum strain) in the fixtures shown in Figure 1. The experimental procedure was to

Table 4
ANELASTIC ENERGY VERSUS THE NUMBER
OF STRAIN CYCLES
(To a 700-MPa Load)

Number of Cycles	Anelastic Energy (MJ/m ³)	E/E ₀ ⁽¹⁾	Number of Cycles	Anelastic Energy (MJ/m ³)	E/E ₀ ⁽¹⁾
Specimen 1 ⁽²⁾			Specimen 2 ⁽³⁾		
4	816.7	1.0	5	699.3	1.0
30	585	0.716	83	542	0.776
72	479	0.586	95	415	0.593
258	333	0.408	185	398	0.569
480	313	0.383	275	373	0.532
942	251	0.306	365	360	0.514
1224	199	0.244	725	209	0.299
1602	187	0.228	1085	198	0.284
1896	172	0.210	1445	171	0.245
2238	151	0.185	1805	160	0.228
			2165	154	0.220
Specimen 3 ⁽⁴⁾			Specimen 4 ⁽⁵⁾		
3	626	1.0	2	887	1.0
30	419	0.668	240	539	0.608
600	352	0.563	540	406	0.457
900	282	0.451	840	373	0.421
1500	259	0.414	1140	313	0.353
2100	216	0.342	1740	284	0.320
			2460	233	0.262

(1) E/E₀ - Anelasticity/Initial Anelasticity.

(2) Solution annealed in argon at 800° C for one hour, water quenched, then prestrained 2.5%.

(3) Solution annealed in argon at 800° C for one hour, water quenched, then prestrained 5.0%.

(4) Solution annealed in a salt bath at 800° C for ten hours, water quenched, then prestrained 2.5%.

(5) Solution annealed in a salt bath at 800° C for ten hours, water quenched, then prestrained 5.0%.

bend a strip, remove the ram, and immerse the female die containing the part into the aging medium (silicone oil for temperatures up to 250° C and lead-bismuth alloy for temperatures above 250° C). After aging, the assembly was quenched to room temperature in still oil and the part removed and measured.

The springback angle was determined by tracing the part contour on paper and measuring the included angle with a protractor. Springback was calculated as:

$$\text{Percent Springback} = \left(1 - \frac{\text{Final Bending Angle}}{180^\circ} \right) 100 .$$

Positive springback means that the part deflected outward when removed from the die; a negative value indicates that the part closed (ie, became loose in the die). Aging temperatures and times varied from 100 to 350° C and from one minute to 100 hours.

Results of this investigation are summarized in Table 5 and plotted in Figure 10. The appearance of the bent specimens, both unaged and aged, can be seen in Figure 11.

Elastic Bending Studies

Strips described earlier were bent, as represented schematically in Figure 3. Twelve specimens were four-point bent to a deflection of 7.8 mm, or an outer fiber stress of 180 MPa (assuming a Young's modulus of 55 GPa), and 12 were bent to a 15.5 mm deflection (360 MPa outer fiber stress). Both of the bend stresses are well below the yield stress of the material. The specimens were then aged in the bent condition in a silicone oil bath for times ranging from one minute to one hour at 150° C, after which the strips were removed from the bend fixture. The results are tabulated in Table 6 and plotted in Figure 12. Deflections were measured with a dial gage and surface plate. Following constrained aging, the specimens were aged unconstrained for an additional 35 hours at 150° C. The results of this aging treatment are reported in Table 6 and portrayed by the graphs of Figures 13 through 18.

DISCUSSION OF RESULTS

Tensile Studies

The results of the 2%-strain-increment tests indicate that:

1. For either the annealed or annealed-plus-aged conditions, U-7.5 Nb-2.5 Zr alloy must be strained approximately 2% before any permanent set occurs. The 2% total extension yield strength was 620 - 690 MPa for annealed material, depending on the quench severity. (A severe quench produces a lower yield strength than does a slack quench.)

The high ratio of yield strength to modulus causes the alloy to exhibit a very large elastic springback.

2. Hydrogen-charged specimens generally had higher yield strengths than vacuum-annealed material. Evidently the lattice is somewhat strengthened by interstitial solution.

Figure 7. PHOTOGRAPHS OF LOAD VERSUS STRAIN FOR A SPECIMEN THAT HAD BEEN ARGON ANNEALED AT 800° C, WATER QUENCHED, THEN PRESTRAINED 2.5% THROUGH 4, 30, 72, 258, 480, 942, 1224, 1602, 1896, 2183, AND 2238 CYCLES.

Figure 8. ANELASTIC ENERGY PER CYCLE AS A FUNCTION OF THE NUMBER OF 100 ksi STRESS CYCLES.

Figure 9. ANELASTIC ENERGY PER CYCLE/INITIAL ANELASTIC ENERGY PER CYCLE AS A FUNCTION OF THE NUMBER OF 100 ksi STRESS CYCLES.

However, there was no evidence of any yield-point phenomenon. Strain aging, which might be expected, did not occur.

3. No strain-rate sensitivity was found for the range of crosshead velocities used in these tests.

4. A "knee" in the elastic region was found in all cases in the third, fourth, and fifth loading cycles for both annealed and annealed-plus-aged materials (see Figures 4 and 5). No knee was evident in the initial and second loading cycles for material annealed plus aged at 150° C (see Figure 4). Instead, the tangent modulus decreased continuously with increasing stress. The knee appears only after plastic deformation has occurred in the material. However, for the vacuum-annealed material aged at 200° C, a second modulus was found in the initial and second loading cycles, but the knee was not as pronounced as it was in subsequent cycles. In addition, except for initial loading, nearly all specimens showed a second change in modulus—approximately 10 to 15 ksi before yielding. This third modulus approximates the average modulus for each loading cycle. It perhaps reflects what the true modulus of the alloy should be if there was no anelastic behavior. Typical values for the three moduli are 58, 30, and 35 GPa, respectively, with knees occurring at approximately 280 and 690 MPa [taken from the third (6%) cycle for vacuum-annealed material]. In all cases, the lower modulus decreased, the middle modulus increased, and the upper modulus remained essentially constant for successive loading cycles, approaching a common value with increasing strain. The knee stress decreased with continued loading, but not always linearly. Aging increased the stress at which the knee occurred.

Table 5
PERCENT SPRINGBACK VERSUS
THE AGING TIME
(180-Degree U Bends to 12.5-mm Radius;
Aging Restrained in the Fixture)

Temperature (° C)	Specimen Number	Springback (%)	Time (hr)
100	64	23.1	0.1
	65	18.2	0.5
	59	16.1	1.0
	60	16.6	5.0
	61	10.3	25.0
	62	11.3	50.0
	63	12.2	100.0
	53	19.5	0.0167
	54	17.4	0.033
	55	16.5	0.083
150	56	16.8	0.167
	57	13.7	0.25
	58	11.5	0.33
	13	12.2	0.5
	3	9.6	1.0
	11	9.2	2.0
	12	7.8	5.0
	48	3.9	15.0
	46	0.1	20.0
	16	0.4	25.0
200	47	0.0	50.0
	49	1.0	100.0
	25	5.9	0.25
	26	2.5	0.50
	28	2.7	0.75
	31	3.9	1.00
	34	0.8	2.0
	37	-1.2	5.0
	40	-6.5	26.5
	29	7.4	0.0167
250	27	1.6	0.0833
	30	3.7	0.0167
	32	2.3	0.25
	33	0.6	0.5
	35	-3.0	0.75
	36	-3.1	1.0
	38	-1.4	1.5
	39	-7.0	2.0
	41	-1.1	0.167
	42	-4.0	0.333
300	43	-10.7	0.500
	18	3.7	0.0833
	44	-7.1	0.167
350	45	-11.3	0.500

5. Although the anelastic energy of corresponding load cycles increased with aging, this is primarily due to the increased flow stress on aging and not to an intrinsic increase in anelasticity with aging.

- On subsequent 2% increments (in addition to the first five cycles), the area within the hysteresis loops actually decreased with increasing strain. These data suggested the stress cycling from 0 to 690 MPa then back to 0 MPa.

The 690-MPa stress-cycling experiments showed that:

- The area within the hysteresis loop decreased exponentially (see Table 4 and Figures 7 through 9) with the number of stress cycles. Extrapolation of the data indicates that zero anelasticity would occur at 20,000 to 30,000 stress cycles.
- All three moduli converged to a value between 40 and 50 GPa.

Figure 10. SPRINGBACK AS A FUNCTION OF THE AGING TIME IN THE FIXTURE. (180-Degree U-Bend Specimens)

3. There was little, if any, difference between vacuum-annealed and hydrogen-charged salt-bath-annealed material.

U-Bend (180-Degree) Studies

For all temperatures covered by these tests, springback was exponential with time. In order to compare the relaxation rates at various temperatures, the time necessary for the springback to be reduced to zero was compared for each temperature. Figure 19 provides a plot of this time versus the reciprocal of the aging temperature. The zero-springback times at each temperature were calculated from a least-squares analysis of the data in Table 5 and are indicated by the arrows in Figure 10. Since the data in Figure 10 can be reasonably well fitted to straight lines, the springback recovery rate (or the strain associated with aging) varies inversely with time. Tests from 100 to 250° C have similar slopes, while those at 300 and 350° C appear to have a different slope. This difference could mean a change in mechanism between 250 and 300° C.

131621
Figure 11. UNAGED SPECIMENS (SPECIMENS 21 AND 23) AND AGED SPECIMENS, SHOWING BOTH POSITIVE AND NEGATIVE SPRINGBACK.

Since the data do not fit a straight line on the Arrhenius plot in Figure 19, aging is not a single-diffusion controlled process. Some of this curvature could result from the influence

Figure 12. PERMANENT DEFLECTION AS A FUNCTION OF THE AGING TIME ON THE BEND FIXTURE. (Elastically Bent Specimens)

of the heat-treating media for the higher-temperature points and extrapolation error for the 100° C point.

To determine whether specimens aged under constraint were stress relieved, either the inner or the outer surfaces of several specimens in both the unaged and aged conditions were removed by electropolishing. If a significant system of residual stresses existed in the specimens, the parts would deflect in response to the removal of some of the surface material. Annealed specimens bent and immediately released springback approximately 35%. On removing the first 0.12 mm from the outer

Figure 13. DEFLECTION OF MULBERRY STRIPS THAT WERE AGED FOR ONE MINUTE AT 150° C ON A FIXTURE, THEN RELEASED AND AGED UNCONSTRAINED AT 150° C.

Table 6
ELASTICALLY DEFORMED SPECIMENS
(Deflection Versus Aging Time at 150° C)

Specimen ⁽¹⁾	Initial Deflection Before Aging (mm)	Initial Aging Time (min)	Deflection after Initial Aging (mm)	Deflection after Aging Unconstrained Specimens (aging time in hours at 150° C) ⁽²⁾				
				1	2	5	10	35
1	7.8	1	5.0	4.5	4.8	5.0	5.2	5.8
2	7.8	1	2.2	0.78	1.0	1.2	1.3	1.6
3	16	1	7.5	7.0	7.8	9.0	9.5	10.5
4	16	1	5.2	3.5	4.0	4.8	4.8	5.5
5	7.8	2	2.0	0.475	0.625	0.68	0.68	0.98
6	7.8	2	5.0	5.2	5.8	6.0	6.0	6.8
7	16	2	7.8	6.2	7.0	7.8	8.0	9.0
8	16	2	7.5	6.8	7.2	7.8	8.0	9.0
9	7.8	6	7.8	8.8	9.0	9.8	9.8	11
10	7.8	6	3.2	2.2	2.6	3.45	2.675	4.55
11	16	6	12	12	13	14	15	16
12	16	6	11	12	12	14	14	15
13	7.8	15	7.8	8.8	9.2	10	10	11
14	7.8	15	8.2	9.0	9.5	10	10	12
15	16	15	16	17	18	20	20	22
16	16	15	14	15	16	16	20	18
17	7.8	30	9.2	9.2	10	12	17	14
18	7.8	30	6.5	7.5	8.0	8.8	9.2	11
19	16	30	18	20	20	21	21	23
20	16	30	16	17	18	20	21	23
21	7.8	60	8.5	9.0	10	10	10	12
22	7.8	60	7.5	7.8	8.5	9.5	9.8	12
23	16	60	16	16	18	19	20	22
24	16	60	16	16	17	19	20	22

(1) Values for the specimen outer fiber stress and strain for the initial deflections, h , used in this study were: initial deflection, h , 7.8 - 16 mm; outer fiber stress, 180 - 360 MPa; outer fiber strain, 0.0033 - 0.0065 mm/mm. Calculations were made according to Elliott,⁽³⁾ using a Young's modulus of 55.1 GPa.

(2) All values in mm.

Figure 14. DEFLECTION OF MULBERRY STRIPS THAT WERE AGED FOR TWO MINUTES AT 150°C ON A FIXTURE, THEN RELEASED AND AGED UNCONSTRAINED AT 150°C .

Figure 15. DEFLECTION OF MULBERRY STRIPS THAT WERE AGED FOR SIX MINUTES AT 150°C ON A FIXTURE, THEN RELEASED AND AGED UNCONSTRAINED AT 150°C .

surfaces, the parts deflected outward an additional 5% (~ 11 degrees). No additional deflection occurred with further surface removal. This result shows the expected compressive residual stress in the outer fibers. No deflections occurred on etching for any of the specimens aged in the fixture at times and temperatures which yielded 0% springback (a maximum of 0.62 mm was removed on either the inner or the outer surface). Evidently, constrained aging can, indeed, relieve residual stresses in plastically deformed specimens.

Metallographic results on Specimen 39, which was aged for two hours at 250°C (-7.0% springback), showed the twin-like structure seen in Figure 20. Knoop indentations were made both on and between the lenticular features in the specimen. There was no perceptible difference in the hardnesses of these areas. The average Knoop hardness was 213.

Figure 16. DEFLECTION OF MULBERRY STRIPS THAT WERE AGED FOR 15 MINUTES AT 150°C ON A FIXTURE, THEN RELEASED AND AGED UNCONSTRAINED AT 150°C .

Figure 17. DEFLECTION OF MULBERRY STRIPS THAT WERE AGED FOR 30 MINUTES AT 150°C ON A FIXTURE, THEN RELEASED AND AGED UNCONSTRAINED AT 150°C .

Elastic Bending Studies

Table 6 and Figure 12 show that, for both 180 and 360 MPa initial bend loadings, all of the deflection associated with these loadings remains in the specimens after only 15 minutes of aging at 150° C. This behavior reveals that this alloy is extremely sensitive to residual stresses and probably explains, to some extent, the dimensional instability that appears during low-temperature aging. The specimens continue to deflect beyond the imposed deflection on further aging. Even after the strips were removed from the bend fixtures and aged, they continued to deflect in the direction of the imposed bend, although those specimens aged for less than 15 minutes did open somewhat before they began to close again (see Table 6 and Figures 13 through 18).

There was considerable scatter in the initial loading deflections. Since the bending apparatus (shown in Figure 2) consisted of two specimen strips bolted together at the ends, and separated

Figure 19. AGING TIME FOR ZERO PERCENT SPRINGBACK AS A FUNCTION OF THE AGING TEMPERATURE.

Figure 18. DEFLECTION OF MULBERRY STRIPS THAT WERE AGED FOR ONE HOUR AT 150° C ON A FIXTURE, THEN RELEASED AND AGED UNCONSTRAINED AT 150° C.

Figure 20. PHOTOMICROGRAPH OF SPECIMEN 39, SHOWING A TWIN-LIKE STRUCTURE. (Specimen Aged for Two Hours at 250° C in the Fixture, -7.0% Springback; Cathodically Etched; Bright Field Illumination; 500X)

in the middle by a block, it was possible for one specimen to deflect more than the other. Therefore, one of the two specimens on each jig might have a considerably lower initial stress than the nominal value for the pair. This condition was obviously the case for Specimens 2, 5, and 10. However, these specimens show the same memory for the bend direction as did the other specimens. This relationship is strong evidence for assuming that the threshold stress for this phenomenon is quite low.

To determine whether elastically bent and aged specimens had been stress relieved, either the inner or outer surface was removed by electropolishing. As in the case of the 180-degree U-bend specimens, no significant deflections were observed, indicating that the specimens were indeed stress relieved.

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