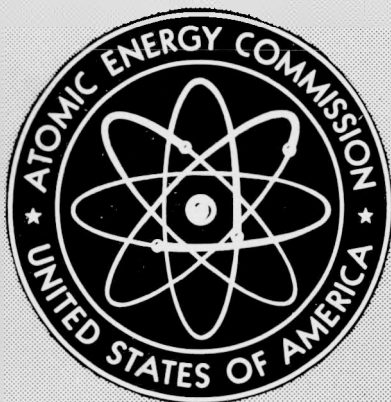


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HIGH STRENGTH ZIRCONIUM-BASE ALLOYS

By
G. A. Henrikson

February 22, 1961

Nuclear Metals, Inc.
Concord, Massachusetts

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High Strength Zirconium-Base Alloys

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February 22, 1961

Nuclear Metals, Inc.
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ABSTRACT

The purpose of this program was to evaluate the mechanical and corrosion properties of certain high-strength zirconium alloys for possible use as structural or fuel element cladding materials. The zirconium alloys studied were prepared by double consumable electrode arc melting followed by extrusion and included:

w/o

Zr - 3 Mo - 2 Al
 Zr - 5 Mo - 2 Al
 Zr - 7 Mo - 2 Al
 Zr - 1 Mo - 5 Al
 Zr - 3 Mo - 5 Al
 Zr - 7 Sn - 1.5 Al
 Zr - 7 Sn - 2.5 Al

Tensile measurements at room temperature were made on these alloys as-extruded, solution treated, and after aging at 600°C for aging times of 1 to 1000 hours. In addition, tensile measurements were made at 600°C on samples aged for 1000 hours at 600°C. Corrosion tests in 680°F water (6 days) and 750°F steam (3 days) were made on as-extruded samples. Room temperature yield strength values (0.2% offset) were: as-extruded material, 130,000 to 190,000 psi; as solution-treated, 80,000 to 140,000 psi; and as-aged at 600°C, 105,000 to 165,000 psi. The elongations at fracture for these materials ranged as follows: as-extruded, 0 to 13 percent; as solution-treated, 0 to 17 percent; and as-aged at 600°C, 0 to 17 percent. The yield strength values obtained at 600°C on samples aged for 1000 hours at 600°C varied between 42,000 and 52,000 psi with elongations at fracture ranging from 15 to 45 percent. The Zr - 5 ^{w/o} Mo - 2 ^{w/o} Al and Zr - 7 ^{w/o} Mo - 2 ^{w/o} Al alloys exhibited some corrosion resistance, whereas the other alloys spalled badly under the test conditions.

I. INTRODUCTION

A. Purpose of Work

The swelling of uranium fuel elements after high burn-up at elevated temperatures is a serious obstacle to the attainment of economical nuclear power. A cladding material which retains its strength at elevated temperatures would be expected to restrain swelling. The purpose of this program was to evaluate zirconium alloys for possible use as such a cladding material.

B. Selection of Alloys

A search of the literature led to a decision to explore simultaneously alloys in two categories:

1. Heat treatable alloys
 - a. "Peritectoid-eutectoid hybrid" alloys
 - b. Alloys containing primarily beta-stabilizers
2. Alpha stabilized alloys

A total of seven zirconium-base alloys were selected, as discussed below, to illustrate the advantages of each class.

1. Heat Treatable

- a. "Peritectoid-eutectoid Hybrid"
(Zr - 1 W/o Mo - 5 W/o Al and Zr - 3 W/o Mo -
5 W/o Al)

In metals which undergo an allotropic transformation, the creep strength can be increased by raising the transformation temperature. In zirconium, peritectoid-type alloying additions such as tin and aluminum perform this function. Chubb,⁽¹⁾ at Battelle, developed "peritectoid-eutectoid hybrid" alloys in which the peritectoid-type alloying element is present to stabilize and strengthen the alpha-zirconium solid solution; the eutectoid-type alloying element is present primarily to lower the lower limit of the alpha plus beta phase region, hence reducing hardness at the hot rolling temperature. Secondly, the eutectoid-type

alloying element strengthens the alpha-zirconium solid solution, produces a precipitate in the microstructure, and makes the alloy heat treatable.

The selection of aluminum and molybdenum as the alloying pair was made on the basis of their properties in binary zirconium alloys. The creep strength of zirconium-aluminum alloys increases with increasing aluminum content. Five percent was selected as the alloying addition on the basis of ease of fabricability. Chubb⁽¹⁾ has shown that the creep strength of binary zirconium-molybdenum alloys increases with increasing molybdenum content to 1.2% and then decreases. Thus, the alloys selected, namely Zr - 1^{w/o} Mo - 5^{w/o} Al and Zr - 3^{w/o} Mo - 5^{w/o} Al, were expected to give a good combination of strength and ease of fabrication.

- b. Primarily Beta-stabilized (Zr - 3^{w/o} Mo - 2^{w/o} Al, Zr - 5^{w/o} Mo - 2^{w/o} Al, and Zr - 7^{w/o} Mo - 2^{w/o} Al)

The requirement for a heat treatable zirconium alloy is that it contain a certain amount of one or more beta stabilizing elements. In addition, strength will be enhanced by additions of alpha-stabilizers. Only molybdenum, niobium and tantalum, when present in sufficient amounts, are known to stabilize beta-zirconium to room temperature during rapid cooling from the beta field. Therefore, one of these elements, when present in smaller amounts, would be expected to produce a heat treatable alloy. Aluminum and tin are both effective alpha stabilizers, with aluminum the most potent strengthener at temperatures below 650°C. An earlier program at NMI demonstrated the feasibility of this type of alloy.⁽²⁾

2. Alpha Stabilized (Zr - 7^{w/o} Sn - 1.5^{w/o} Al and Zr - 7^{w/o} Sn - 2.5^{w/o} Al)

Van Thyne and McPherson⁽³⁾ at Armour developed alloys in which the additions stabilized alpha-zirconium. Such alloys may be expected to possess high temperature properties similar to titanium-base alloys.

In general, the tin-aluminum alloying additions retained strength considerably better than any other additions for temperatures to 650°C.

Above this temperature, the zirconium-tin binary alloys were superior in short-time tensile testing, the only test reported. This leads to the conclusion that aluminum is a more effective strengthener at lower temperatures and tin the more favorable addition for high temperatures.

The zirconium-aluminum-tin ternary alloys were hard to forge and heavy scaling was encountered, but canning to reduce oxidation and then extruding at high temperature overcomes these difficulties. It was also thought that the tin content might aid in resistance to corrosion by water.

C. Methods of Evaluation

Material for experimental study was prepared by the extrusion of consumable-electrode vacuum arc-melted alloys. Vacuum arc-melting was employed to obtain homogeneous melts with a minimum of contamination. Effects of the alloying additions on the alpha-beta transformation temperature were studied by dilatometry, where possible, as a guide to heat treating temperatures. The scope of this program did not permit any x-ray or metallographic examinations of the structural changes, although these tests would have proven helpful in the alloys for which a transformation temperature could not be determined.

Evaluations of the alloys were carried out by tensile and corrosion testing. Tensile tests were conducted at room temperature on material in the as-extruded, as-solution treated, and the as-solution treated and aged conditions. In addition, tensile testing was done at 600°C on material aged for 1000 hours. The corrosion testing was carried out in water at 680°F and steam at 750°F, which are environments encountered in nuclear reactors.

All alloys, including those containing alpha-stabilizers only, were compared for response to solutionizing and aging treatments. It was not possible to perform exhaustive heat treating studies. Consequently, it is not known whether optimum properties have been achieved for any of the alloys.

II. EXPERIMENTAL PROCEDURES

A. Materials

All alloys used in the program were obtained as 35 pound billets, 3.85-inch diameter by 12 inches long, from Oregon Metallurgical Corp. The ingots were prepared by double consumable-electrode melting under vacuum. Reactor grade zirconium sponge was used. The purest materials commercially obtainable were used for the alloying additions. Chemical analyses of the ingots are given in Table I.

B. Fabrication

1. Extrusion of Rods

Approximately 4 inches of each billet was extruded into 0.600-inch diameter rods for subsequent machining to the desired test specimens. For the primary extrusion, 16-gage copper cans were used for the aluminum-molybdenum alloys and 16-gage steel for the remaining alloys. For all alloys except the 3^{w/o} Mo - 2^{w/o} Al and 5^{w/o} Mo - 2^{w/o} Al alloys, two extrusions were needed to obtain the size required for machining. Complete data for the extrusions are given in Tables II and III. Alundum powder was used as the parting compound between can and billet for all the extrusions.

2. Machining of Test Specimens

The major portion of each rod was machined into standard ASTM miniature tensile testing specimens with 1/2-20 NF threads and a 1-inch gage length of 0.350-inch diameter. The remainder of the extruded rod was machined into dilatometer and corrosion study specimens. The dilatometer specimens were 2 inches long and 1/4 inch in diameter with a 0.093-inch hole 1/4-inch deep in one end. For the corrosion studies, cylinders 1/2-inch diameter by 1/2-inch long were machined.

C. Dilatometry

The dilatometer specimens were subjected to an homogenization heat treatment prior to use. All samples were sealed in vycor under an argon atmosphere and held 96 hours at 1000°C, water quenched to room temperature, resealed in vycor, held 240 hours at 600°C, and water quenched to room temperature.

In the dilatometer, the specimen was under vacuum with the instrument itself designed to plot elongation vs temperature on an X-Y recorder. Previous probing with five thermocouples spaced 1/2 inch apart indicated uniform heating rates and temperature over the entire specimen length. Dummy runs with a quartz rod in place of the specimen showed no differential elongation at any temperature up to 1200°C.

D. Solution Heat Treatments and Aging Studies

The tensile specimens from each alloy were given a solution heat treatment prior to aging. The solution heat treatments were performed under dynamic vacuum at a temperature 50°C above the alpha-to-beta transformation temperature. The samples were heated to this temperature, held for 2 hours, and quenched in water at room temperature. For the alloys for which there were no dilatometer results, the solutionizing temperature was deduced from the available phase diagrams. The solutionizing temperatures used are given in Table IV.

After the solution treatment, the tensile specimens were aged at 600°C for periods from 1 to 1000 hours. For aging periods of 1, 3, 10, and 30 hours, samples were heated in vacuum and were quenched directly in room temperature silicone oil while still under the dynamic vacuum. The furnace design allowed the treatment of eight specimens with one loading. For aging times of 100, 300, and 1000 hours, the specimens were sealed in vycor tubes under one-third atmosphere argon, heated to 600°C for the appropriate time, and quenched in oil.

In addition to the room temperature tests above, a group of samples was aged 1000 hours and tested at 600°C.

E. Tensile Testing

The tensile testing was performed by the New England Materials Laboratory, Inc. A Baldwin-Lima-Hamilton 60,000 pound testing machine was used at a strain rate of 0.05 in./in./min. A stress-strain recording was obtained for each test. The high-temperature testing was done in air with the samples heated in a split-muffle furnace mounted on the cross head of the tensile machine.

F. Corrosion Testing

Corrosion testing of as-extruded samples was carried out under the usual conditions for Zircaloy (680°F water and 750°F steam).

III. RESULTS AND DISCUSSION

The dilatometry technique was successful in determining the transformations in the 3^{w/o} Mo - 2^{w/o} Al, 5^{w/o} Mo - 2^{w/o} Al and the 7^{w/o} Sn - 1.5^{w/o} Al alloys, but unsuccessful with the others, and additional work could not be carried out within the scope of this program. The transformation temperatures of the alloys listed above are given in Table V.

For this program, only the transformation temperatures during heating were of major importance. A complete heating-cooling cycle was made for each of the alloys successfully run and the data showed that the transformations on cooling were as pronounced as those on heating, but that they occurred at lower temperatures. No evidence of hysteresis was found when the cooling cycle was carried to temperatures below 500°C. It may be that the unsuccessful runs were due to sluggish transformations of the alloys or transformation temperatures higher than 1150°C, the maximum temperature at which reliability of readings was assured.

The tensile properties of the as-extruded alloys are given in Table VI. Yield strengths as high as 191,000 psi and elongations as high as 12% were obtained. Tensile properties of the as-solution treated alloys are given in Table VII. Yield strengths of 140,000 psi and elongations

of 16% are noted. Ultimate strengths and yield strengths both decreased in the solution-treated alloys. In general, the decrease in yield strength is more pronounced than the decrease in ultimate tensile strength. Elongations showed a general, but less consistent, tendency to increase. This behavior is expected because of the difference in cooling rates for the two treatments. Some transformation to the harder alpha-phase undoubtedly occurs during the air cooling which follows extrusion.

In obtaining tensile data on aged material, many of the tests were invalidated when the specimens broke with elongations less than the 0.2% required for yield strength determinations. In addition, a number of specimens broke in the threads, indicating that the notch sensitivity is high. This trouble did not show up with either the as-extruded or the as-solution treated samples. Complete results of the aging studies, available for only three of the alloys, are tabulated in Table VIII and given graphically in Figs. 1 and 2. It is shown that the aging behavior of the alpha-stabilized alloys (7.5 Sn - 1.5 Al and 7.5 Sn - 2.5 Al) results in an increase in the yield strength with time, but little change in the ultimate strength. The alloy containing the beta stabilizer, molybdenum, behaved as expected, with the yield strength reaching a maximum in a relatively short time and then declining as overaging takes place. No metallographic observations were made to determine if any visible structural changes had taken place. In addition to the 600°C aging study, a number of samples were aged at 500°C, but the premature failure problem left too few samples to draw any conclusions as to effects of aging temperature.

Results of the tensile tests at 600°C are given in Table IX. Of special interest is the difference in elongation between the alloys containing alpha stabilizers only and those containing beta stabilizers. The alloys containing beta stabilizers exhibit two to three times as much elongation as the alloys containing alpha stabilizers only. The yield strength values, however, do not show this marked differentiation.

In Table X, additional tensile data for the 3^{w/o} Mo - 2^{w/o} Al alloy are combined with data from Tables VI, VII and VIII for this alloy. Of significance is the decrease in the yield strength of the as-extruded

material after aging 30 hours at 600°C to the same level as the solution-treated material. Comparison with the material aged at 500°C suggests that the 600°C aging temperature used was too high and resulted in rapid overaging of the alloy.

Corrosion test results (Table XI) show that only the 5^{w/o} Mo - 2^{w/o} Al and 7^{w/o} Mo - 2^{w/o} Al alloys have any possibility for use in high temperature water or steam environments. A possible way to overcome this deficiency would be to extrude with a cladding of a more corrosion resistant material.

IV. CONCLUSIONS

1. Zirconium alloys with 600°C strengths significantly higher than stainless steel can be produced by additions of alpha and beta stabilizing elements.

2. The high strength alloys studied here would have considerable strength as a cladding in the as-worked condition and do not require complicated heat treatments. This is evidenced by the high strengths retained in as-extruded material with no attempt made to optimize properties.

3. Ductilities of these alloys are low at room temperature but increase appreciably at elevated temperatures.

4. Corrosion behavior of the alloys precludes their use in high temperature water or steam without additional protection.

Table I
Chemical Analyses of Ingots

Alloy (w/o)	Composition in w/o								Composition in ppm*			
	Mo		Al		Sn		N		Hf		Fe	
	T	B	T	B	T	B	T	B	T	B	T	B
3 Mo-2 Al	3.32	2.70	2.06	1.59			0.003	0.004	<100	<100	860	850
5 Mo-2 Al	5.27	5.00	1.98	2.03			0.002	0.003	<100	<100	850	890
7 Mo-2 Al	6.60	6.84	1.98	1.86			0.007	0.011	150	<100	1200	900
1 Mo-5 Al	0.90	1.05	4.61	4.85			0.006	0.010	<100	<100	900	920
3 Mo-5 Al	3.20	3.20	4.92	4.86			0.006	0.012	<100	<100	850	900
7 Sn-1.5 Al			1.47	1.37	6.98	7.46	0.002	0.003	66	71	880	1150
7 Sn-2.5 Al			2.78	2.36	7.03	7.36	0.009	0.007	40	40	2050	2740

*Spectrographic analyses for B, Cd, Co, Cr, Cu, Mg, Mn, Ni, Pb, Si, Ti, V, and W indicated less than 100 ppm for any of these.

T = Top; B = Bottom.

Table II

Data for Primary Breakdown Extrusions*

Alloy (^{w/o})	3 Mo-2 Al	5 Mo-2 Al	7 Mo-2 Al	1 Mo-5 Al	3 Mo-5 Al	7 Sn-1.5 Al	7 Sn-2 5 Al
Can	16 gage copper	16 gage copper	16 gage copper	16 gage steel	16 gage steel	16 gage steel	16 gage steel
Cutoff	copper	copper	graphite	graphite	graphite	graphite	graphite
Billet Temp. (°F)	1550	1550	1575	1832	1832	1832	1832
Die Size (in.)	0.600	0.600	2.000	2.000	2.000	2.000	2.000
Reduction	45.6:1	45.6:1	4:1	4:1	4:1	4:1	4:1
Extrusion Constant (psi)	40,700	37,100	48,200	34,200	33,700	44,200	44,900

*For all alloys:

Cone: mild steel
 Evacuation: none
 Liner Size (in.): 4.050
 Liner Temp. (°F): 900
 Lubricant: Oildag
 Ram Speed: 14 in./min

Table IIIData for Secondary Extrusions*

Alloy (^w /o)	7 Mo-2 Al	1 Mo-5 Al	3 Mo-5 Al	7 Sn-1.5 Al	7 Sn-2.5 Al
Billet Temp. (^o F)	1575	1832	1832	1832	1832
Extrusion Constant (psi)	47,500	40,200	39,800	50,100	43,800

*For all alloys:

Can: 16 gage steel
Cone: mild steel
Cutoff: graphite
Evacuation: none
Liner size (in.): 2.040
Liner Temp. (^oF): 900
Die Size (in.) 0.600
Reduction: 11:1
Lubricant: Oildag
Ram Speed: 30 in./min

Table IVSolution Heat Treatment Temperatures

Alloy (w/o)	Temp. (°C)
3 Mo-2 Al	800
5 Mo-2 Al	850
7 Mo-2 Al	850
1 Mo-5 Al	1000
3 Mo-5 Al	1000
7 Sn-1.5 Al	1090
7 Sn-2.5 Al	1090

Table VAlpha-to-Beta Transformation Temperatures
of Alloys on Heating (from dilatometer)

Alloy (w/o)	Transformation Temp. (°C)	
	Start	Finish
3 Mo-2 Al	700	750
5 Mo-2 Al	720	800
7 Sn-1.5 Al	965	1050

Table VITensile Properties of Alloys As-extruded

Alloy (w/o)	Yield	Ultimate	Elongation (%)
3 Mo-2 Al	147,500	178,500	10.8
5 Mo-2 Al	145,000	160,000	4.2
7 Mo-2 Al	136,200	147,700	8.7
1 Mo-5 Al	144,000	162,000	12.9
3 Mo-5 Al	191,500	218,500	*
7 Sn-1.5 Al	129,800	143,800	2.6
7 Sn-2.5 Al	163,000	176,000	3.9

*Very low elongation; measurement could not be made because of breakup of specimen.

Table VIITensile Properties of Alloys As-solution Treated

Alloy (w/o)	Yield	Ultimate	Elongation (%)
3 Mo-2 Al	107,000	141,500	16.8
5 Mo-2 Al	93,500	139,700	11.4
7 Mo-2 Al	120,000	143,200	11.8
1 Mo-5 Al	79,200	165,000	11.7
3 Mo-5 Al	*	*	*
7 Sn-1.5 Al	106,000	131,000	5.5
7 Sn-2.5 Al	141,300	158,700	1.9

*Measurement could not be made because of premature failure of sample in threads.

Table VIII
Aging Data for Solution Treated Alloys

Aging Time at 600°C (hr)	Yield Strength (psi)	Ultimate Strength (psi)	Elongation (%)
<u>7 Sn-2.5 Al</u>			
0	141,000	158,700	1.9
1	148,000	165,000	0.2
3	145,000	163,000	*
10	148,000	162,000	*
30	157,000	167,000	*
100	159,400	173,000	*
300	162,000	176,000	*
1000	164,000	175,000	*
<u>7 Sn-1.5 Al</u>			
0	106,000	131,000	5.5
1	110,500	133,000	5.5
3	113,000	139,000	4.4
10	118,500	137,000	3.1
30	115,500	139,000	0.8
100	119,000	132,000	*
300	123,500	135,700	*
1000	124,000	130,000	*
<u>3 Mo-2 Al</u>			
0	107,000	141,500	16.8
1	130,000	143,500	14.6
3	129,000	146,500	16.7
10	127,000	143,300	13.4
30	125,000	142,500	10.2
100	112,500	136,000	4.0
300	110,000	132,500	7.0
1000	94,500	120,500	6.0

*Not measurable because of breakup of specimen.

Table IX
Tensile Properties at 600°C*

Alloy (^{w/o})	Yield	Ultimate	Elongation (%)
3 Mo-2 Al	43,600	57,500	33.3
5 Mo-2 Al	48,200	68,300	34.4
7 Mo-2 Al	45,900	76,000	36.2
1 Mo-5 Al	46,600	75,600	29.0
3 Mo-5 Al	42,500	73,700	45.0
7 Sn-1.5 Al	51,500	65,500	15.2
7 Sn-2.5 Al	47,400	75,000	16.6

*Solutionized and aged at 600°C for 1000 hours prior to test. Test made in air.

Table X
Additional Tensile Data for 3 Mo-2 Al Alloy

Treatment	Yield Strength (psi)	Ultimate Strength (psi)	Elongation (%)
As-extruded	147,500	178,500	10.8
As-extruded and aged 30 hr at 600°C	128,000	134,000	*
As-solution treated	107,000	141,500	16.8
As-solution treated and aged 30 hr at 500°C	138,700	155,000	*
As-solution treated and aged 30 hr at 600°C	125,000	142,500	10.2

*Not measurable because of breakup of specimen.

Table XI

Corrosion Results of As-extruded Stock

Alloy (^{w/o})	680°F Water for 6 Days	750°F Steam for 3 Days
3 Mo-2 Al	Spalling badly	Spalling badly
5 Mo-2 Al	Shiny, adherent gray-black oxide film except spalling where scribed	Shiny, adherent gray-black oxide film except spalling where scribed
7 Mo-2 Al	Shiny, adherent gray-black oxide film (weight gain of 170 mg/dm ²), some spalling after 14 days	Shiny, adherent gray-black oxide film (weight gain of 265 mg/dm ²), spalling badly after 6 days
1 Mo-5 Al	Spalling badly	Spalling badly
3 Mo-5 Al	Spalling badly	Spalling badly
7 Sn-1.5 Al	Spalling badly	Spalling badly
7 Sn-2.5 Al	Spalling badly	Spalling badly

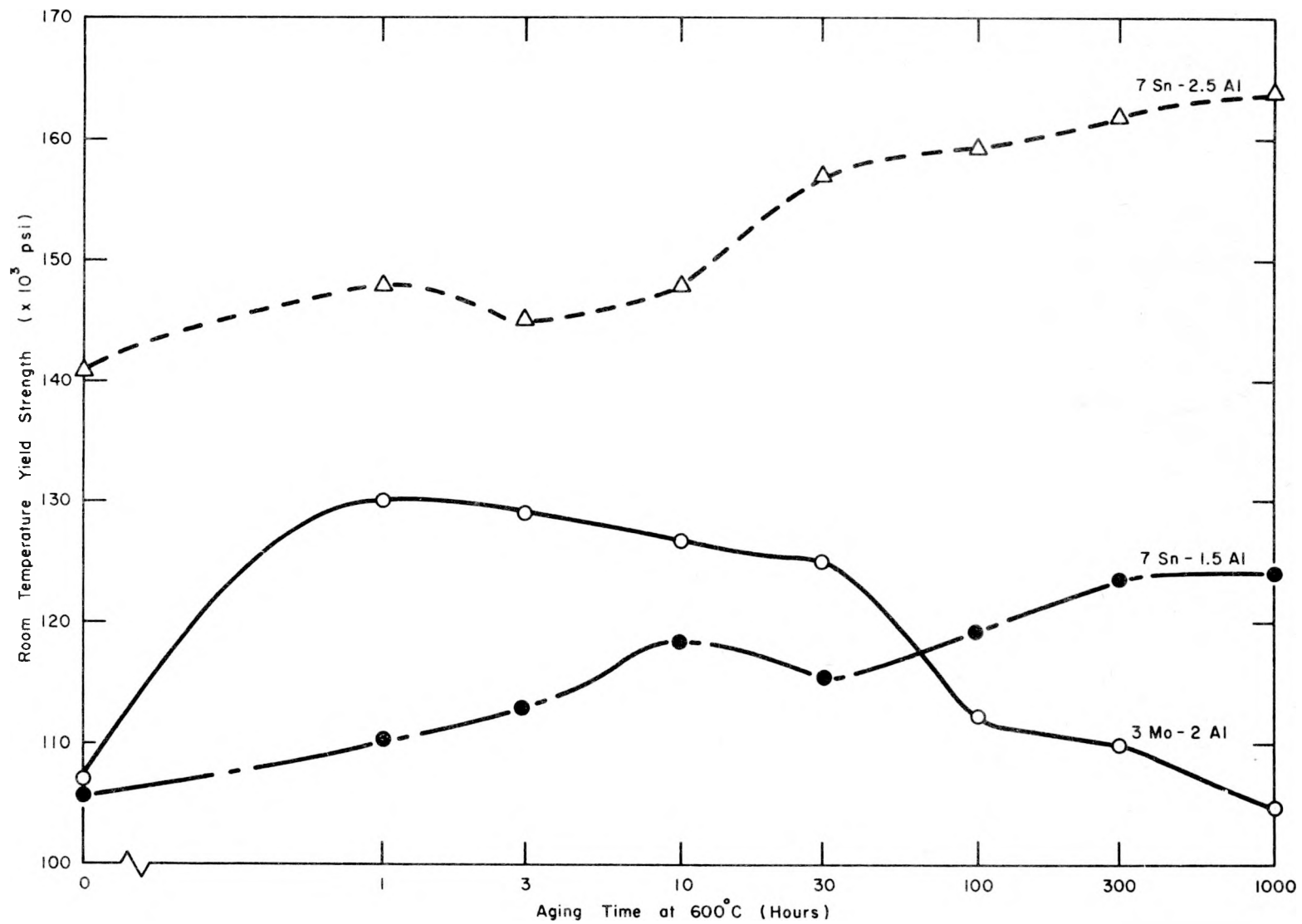


Figure 1. Room temperature yield strength vs. aging time at 600°C .
Drawing No. 1918

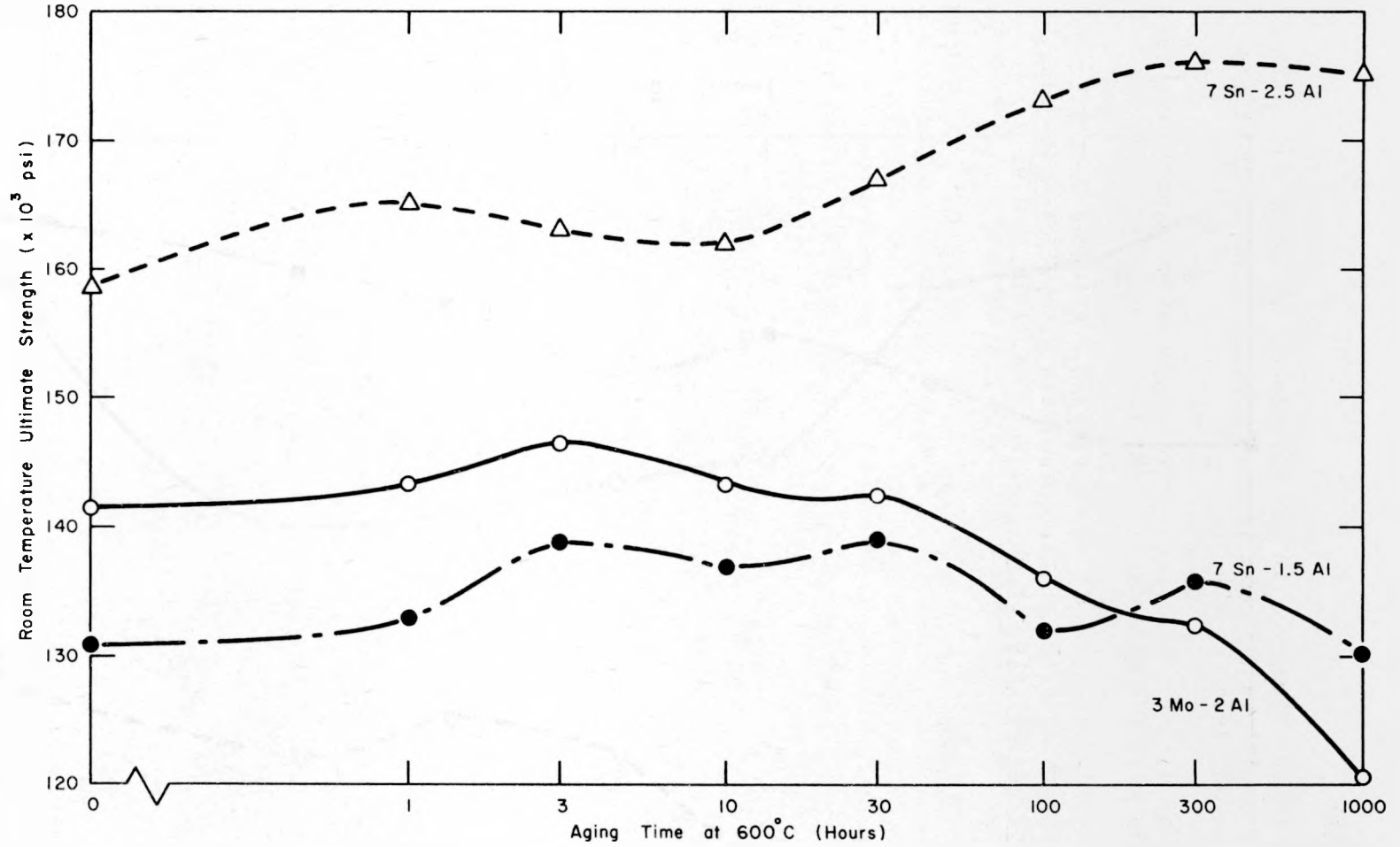


Figure 2. Room temperature ultimate strength vs. aging time at 600°C.
Drawing No. RA-1919

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