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AGING BEHAVIOR OF ALLOY 800H AND ASSOCIATED MECHANICAL PROPERTY CHANGES

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ABSTRACT

Aging behavior of five different heats of Alloy 800H was investigated in the temperature range 538 to 816 C (1000 to 1500 F) for times up to 8,000 h. Significant age-hardening was observed at low temperatures [i.e., 538, 593 and 649 C (1000, 1100 and 1200 F) in most heats. One of the heats, however, exhibited less hardening. Microstructural changes occurring during aging were evaluated by optical metallography, transmission electron microscopy, and x-ray diffraction.

Effects of thermal aging on both tensile properties and impact toughness properties were evaluated. The results show that the alloy exhibits good residual impact toughness and tensile ductility after aging up to 8,000 h.

INTRODUCTION

The high temperature gas-cooled reactor (HTGR) designed by General Atomic Company contains many metallic components that must operate for very long times (up to 40 years) at elevated temperatures [approximately 538 to 816 C (1000 to 1500°F)]. The use of an alloy for these components requires an understanding of structural and mechanical property changes as a result of thermal aging. Thus, as a part of the program in support of the development and design of HTGRs, a systematic investigation of the thermal stability of several high temperature alloys is underway at General Atomic. This paper presents the results of an investigation into the aging behavior and mechanical property changes of Alloy 800H for times up to 8000 hr.

EXPERIMENTAL PROCEDURES

Materials of five different heats in bar form were purchased in the solution-annealed condition. Chemical analyses, as supplied by the vendor, are shown in Table 1. Aging was performed in air at 538, 593, 649, 760 and 816°C (1000, 1100, 1200, 1400 and 1500°F) for times up to 8000 hr. Samples for hardness, microstructural examination and mechanical property testing were obtained from aged bars after removal of surface layers affected by oxidation.

Impact tests were conducted on standard Charpy V-notch specimens in accordance with the requirements of ASTM E23-72. Room-temperature and elevated-temperature tensile tests were conducted in an Instron machine using round specimens with 0.635 cm (0.25 in.) diameter. All tensile tests were carried out in air in accordance with ASTM E21-70.

Fracture surfaces were examined in a scanning electron microscope. Microstructure was characterized by optical metallography, X-ray diffraction, and transmission electron microscopy. The carbide precipitates analyzed by X-ray diffraction were obtained by electrolytically extracting the aged samples using an electrolyte containing 10 pct HCl in methanol. A Guinier De Wolff camera was used in conjunction with CuKα radiation. Thin foils for transmission electron microscopy were prepared by a twin jet electropolisher using an electrolyte of 10% perchloric acid in methanol. The foil was then examined in a Philips EM-300 electron microscope operating at 100 kV.

RESULTS AND DISCUSSION

AGING BEHAVIOR

The aging behavior of five different heats of material was evaluated by observing the hardness changes with time up to 8000 hr at 538, 593, 649, 760 and 816°C (1000, 1100, 1200, 1400 and 1500°F). Aging at high temperatures (i.e., 760 and 816°C) resulted in little change in hardness for times up to 8000 hr for all heats of material. At 538, 593 and 649°C (1000, 1100 and 1200°F) Heats A, B, C and D exhibited similar aging behavior. They showed significant age-hardening at 593 and 649°C (1100 and 1200°F), and intermediate hardening at 538°C (1000°F). There was no evidence of overaging for times up to 8000 hr for the above four heats with the exception of Heat A aged at 649°C (1200°F) which showed a hardness peak at 500 hr. In contrast to the above four heats, Heat E exhibited considerably less hardening at 593°C (1100°F) and no hardening at 649°C (1200°F). Some hardening was observed at 538°C (1000°F). This difference in age-hardening behavior is illustrated in Fig. 1, in which only Heat A data were plotted for comparison with Heat E.

The age-hardening behavior at low temperatures (i.e., 538, 593 and 649°C) was further investigated by conducting tensile tests at room- and elevated-temperatures. Elevated temperature tensile tests were conducted at the respective aging temperatures. Tensile tests were conducted on Heats A and E. The results as shown in Table 2, are in good agreement with the hardness data in that significant increases in yield strength were observed after aging for Heat A, but not for Heat E.

For Heat A which exhibited significant age-hardening, increases in yield strength are considerably more pronounced at elevated-temperatures than at room-temperature. Aging at 593°C (1100°F) for 4000 hr resulted in an increase in room-temperature yield strength by 25%, but by 140% at

593°C (1100°F). Similarly, an increase in yield strength by 20% at room-temperature, whereas by 130% at 649°C (1200°F) was observed after aging at 649°C for 4000 hr. Aging at 538°C (1000°F) for 4000 hr resulted in little increase in room-temperature yield strength, yet a significant increase (by 84%) in yield strength at 538°C. The above is clearly illustrated in Fig. 2.

MICROSTRUCTURE

Specimens of Heat A aged for 8000 hr were examined by thin foil transmission electron microscopy. $M_{23}C_6$ carbides were observed in all specimens. These carbides precipitated mainly at twin and grain boundaries. Intragranular carbides were also observed. They were few and coarse at 760 and 816°C (1400 and 1500°F), fine and extensive at 649°C (1200°F). At 593°C (1100°F) intragranular carbides which precipitated mainly around primary carbides [presumably TiN and/or Ti(C,N)] were observed. The distribution of intragranular carbides after 8000-hr aging is best illustrated in optical photomicrographs as shown in Fig. 3. γ' $[Ni_3(Al,Ti)]$ precipitates were observed only in the specimen aged at 649°C (1200°F). No γ' precipitates were observed in the specimens after aging for 8000 hr at 593°C (significant age-hardening) and at 538°C (intermediate age-hardening).

Strengthening of nickel-based alloys by the precipitation of intermetallic compounds, $\gamma'[Ni_3(Al,Ti)]$, is well known. Numerous papers (Ref. 1 through 6) have attributed the age-hardening and increased creep strength in Alloy 800 or Alloy 800H to a homogeneous precipitation of γ' precipitates.

In the present study it is easier to account for the hardening at 649°C (1200°F), since extensive γ' precipitates and intragranular carbides were observed. The γ' precipitates were observed to form as early as 96 hr at this temperature, as shown in Fig. 4. To account for the hardening at 538 and 593°C (1000 and 1100°F) becomes much more difficult since no γ' nor extensive intragranular carbides were observed by TEM even after aging for 8000 hr. This led Villagrana, et al. (Ref. 7) to suggest that the hardening was primarily due to grain boundary carbides ($M_{23}C_6$). They (Ref. 7) observed that grain boundary carbides were small and dense at 538, 593 and 649°C, but large and few at 703 and 760°C after aging for 4000 hr.

Early stages of carbide precipitation in Heat A were studied by TEM. Figure 5 shows typical grain boundary carbides after short time aging (336 hr) at 538, 593, 649 and 760 (1000, 1100, 1200 and 1400°F). Only few and small carbides were observed at grain boundaries after aging at 538°C (1000°F) for 336 hr. Aging at 593, 649 and 760°C (1100, 1200 and 1400°F) produced dense grain boundary carbides. The carbides, however, coarsened as the temperature increased. There is no significant difference in the interparticle spacing for these grain boundary carbides aged at the above three temperatures. As shown in Fig. 1, at 336 hr of aging, significant increases in hardness at 593 and 649°C (1100 and 1200°F), but little hardness increase at 760°C (1400°F) were observed. Tensile tests were conducted at 593°C (1100°F) on specimens of Heat A aged at 760°C (1400°F) for 500 hr showing no increase in yield strength as compared to the solution-annealed material. The structure consisting of dense grain boundary carbides obtained by aging at 760°C (1400°F) for approximately 300 to 500 hr resulted in no significant age-hardening (i.e., no increases in room-temperature hardness or yield strength at 593°C). This implies that the dense grain boundary carbides developed at 760°C (1400°F) have no strengthening effects.

Continued aging to 8000 hr at high temperature (760 and 816°C) resulted in coarsening of grain boundary carbides. They became very coarse and far apart. At lower temperatures (593 and 649°C) the grain boundary carbides coarsened extremely slowly and remained dense and extensive after 8000 hr. At 538°C carbides continued precipitating at grain boundaries resulting in fine and dense grain boundary carbides after 8000 hr. The above observation on the morphology of grain boundary carbides after 8000 hr were similar to those observed by Villagrana, et al. (Ref. 7) after 4000-hr aging. It should be noted that significant age-hardening at low temperatures was observed after a short time aging

(∿500 hr). It was considered important, therefore, to study the early stages of precipitation and correlate the observations to the age-hardening behavior.

Carbides were extracted and analyzed by X-ray diffraction for specimens of Heats A and E aged for 500 hr at 538, 593, 649, 760 and 816°C. The results are given in Table 3. Both heats of material contained $M_{23}C_6$, Ti(C,N) and TiN in the solution-annealed condition. Heat E contains larger amounts of $M_{23}C_6$ and TiN precipitates than Heat A in the solution-annealed condition. For both heats of material aged at all temperatures, both Ti(C,N) and TiN remained relatively unchanged, while the amount of $M_{23}C_6$ carbides increased with increasing temperature. The amount of $M_{23}C_6$ was highest when aged at 649, 760 and 816°C for Heat A, and at 760 and 816°C for Heat E.

To further investigate the correlation between carbides and age-hardening, further aging treatment experiments coupled with tensile tests and carbide extraction were performed on specimens of Heats C and E. Three aging treatments were employed: (1) Aging at 593°C (1100°F) for 1000 hr; (2) aging at 760°C (1400°F) for 1000 hr; and (3) aging at 760°C (1400°F) for 1000 hr followed by aging at 593°C (1100°F) for 1000 hr. Tensile tests were then conducted at both room-temperature and at 593°C (1100°F). The results of yield strengths and amounts of carbides extracted are given in Table 4. As shown in the table, aging at 593°C (1100°F) for 1000 hr resulted in an increase in the amount of carbide precipitates for both heats. As discussed previously, this increase due to aging is related to the precipitation of $M_{23}C_6$ carbides. For both heats of material aging at 760°C (1400°F) for 1000 hr produced many more carbides than aging at 593°C (1100°F). An additional 1000 hr at 593°C (1100°F) following aging at 760°C (1400°F) for 1000 hr did not increase the amount of carbides within an experimental error.

No increase in yield strengths at room-temperature and at 593°C (1100°F) was observed for specimens of Heat E after the above three different aging treatments.

For Heat C, aging at 760°C (1400°F) for 1000 hr produced no increase in yield strengths at room-temperature and at 593°C (1100°F). However, aging at 760°C (1400°F) for 1000 hr followed by aging at 593°C (1100°F) for 1000 hr increased the yield strength to the levels comparable to those after aging at 593°C (1100°F) for 1000 hr. This additional aging at 593°C (1100°F) for 1000 hr, which caused little additional carbide precipitation, resulted in a significant increase in the yield strengths at room-temperature and at 593°C (1100°F). The above results again suggest that in the heats studied in this program, there was no apparent correlation between age-hardening and carbide precipitation at 593°C (1100°F).

From overall consideration of the above discussion, it is believed that age-hardening observed at 649°C (1200°F) is primarily due to a homogeneous precipitation of γ' precipitates in the grain interior. Intragranular precipitation of carbides observed at this temperature could also contribute to the hardening. At 538° and 593°C (1000° and 1100°F) the results fail to reveal the possible mechanisms responsible for the observed hardening. No γ' precipitates were observed even after 8000-hr aging at these temperatures in the heat which exhibited age-hardening. No extensive intragranular carbide precipitation was observed which can account for the observed hardening. The only microstructural evolution observed during aging at these temperatures was the precipitation of grain boundary carbides. Nevertheless, the results of the present investigation, with a particular emphasis on the short-time aged specimens, indicate no apparent correlation between age-hardening and the precipitation of grain boundary carbides at these temperatures.

IMPACT TOUGHNESS AND TENSILE DUCTILITY CHANGES

The change of room-temperature impact toughness properties of specimens of Heat A as a function of aging time up to 8000 hr was investigated, and is shown in Fig. 6. All aging temperatures produced a substantial decrease in impact toughness after initial short-time aging, and the toughness tended to level off upon long-time aging. After aging for

8000 hr, the specimens aged at 593 and 649°C (1100 and 1200°F) which produced the highest hardness exhibited the minimum toughness with a predominantly intergranular fracture. A slight recovery of impact toughness was observed at 649°C (1200°F) after aging for 8000 hr. The hardness curves of this heat of material also indicate overaging after 8000 hr exposure at 649, 760 and 816°C (1200, 1400 and 1500°F). Aging at 593°C (1100°F), however, showed a continuous decrease (although small) in impact toughness in conjunction with a gradual increase in hardness after 8000 hr. The specimens with much higher impact toughness when aged at 538, 760 and 816°C (1000, 1400 and 1500°F) exhibited predominantly transgranular dimple rupture. Figure 7 shows fracture surfaces of the Charpy specimens aged for 8000 hr.

Residual tensile ductilities after aging for 500 and 4000 hr were investigated for specimens of Heats A and E; the results are shown in Figs. 8 and 9. Specimens of Heat A aged at 593 and 649°C (1100 and 1200°F) for 4000 hr, which produced significant age-hardening, showed only a slight drop in room-temperature tensile ductility. Aging at 538°C (1000°F), however, improved the room-temperature tensile elongation. A decrease in the elevated-temperature tensile elongation was observed after 4000 hr at 538 and 593°C (1000 and 1100°F). The elevated-temperature tensile elongation first decreased after 500 hr and then increased after 4000 hr during aging at 649°C (1200°F), but was relatively unaffected after aging for 4000 hr at 760°C (1400°F).

As for specimens of Heat E, a slight drop of the room-temperature tensile elongation was observed after 4000 hr at 538, 593, 649, 760 and 816°C (1000, 1100, 1200, 1400 and 1500°F). The elevated-temperature tensile elongation decreased after aging at the temperatures which produced some age-hardening (538 and 593°C), and increased significantly after aging at 649, 760 and 816°C (1200, 1400 and 1500°F) which showed no age-hardening.

SUMMARY & CONCLUSIONS

The aging behaviors of five different commercial heats of Alloy 800 H were investigated at 538°, 593°, 649°, 760° and 816°C (1000°, 1100°, 1200°, 1400° and 1500°F). Significant age-hardening was observed to occur at lower aging temperatures in four of the five heats. The other heat showed lesser age-hardening. Microstructural studies and step aging experiments failed to reveal age-hardening mechanisms responsible for the hardening observed at 538° and 593°C. Age-hardening observed at 649°C is believed to be due to a homogeneous precipitation of γ' precipitates in the grain interior. Intragranular precipitation of carbides observed at this temperature could also contribute to the hardening.

The alloy retained good tensile ductility after aging. In addition, Charpy impact tests on specimens from an age-hardenable heat showed that the material retains good residual room-temperature impact toughness after aging for 8000 hr in the temperature range of 538 to 816°C (1000 to 1500°F).

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TABLE 1
CHEMICAL COMPOSITION AND GRAIN SIZE OF EACH HEAT

Heat	Chemical Composition (wt %)(a)										Grain Size ASTM
	C	Si	Mn	Cu	Fe	Cr	Ni	Ti	Al	S	
A	0.05	0.26	0.49	0.42	42.75	20.23	34.94	0.44	0.39	0.007	3
B	0.07	0.24	0.90	0.52	43.98	20.34	33.03	0.42	0.47	0.007	5
C	0.07	0.39	0.77	0.49	43.60	20.36	33.43	0.46	0.43	0.004	4
D	0.07	0.32	0.80	0.53	45.93	20.09	31.65	0.35	0.25	0.006	4
E	0.08	0.41	0.93	0.16	45.21	20.63	31.97	0.26	0.32	0.007	4

(a) Supplied by vendor.

TABLE 2
ROOM-AND ELEVATED-TEMPERATURE YIELD STRENGTHS OF HEATS A AND E AFTER 4000-HR AGING IN COMPARISON WITH THOSE OF SOLUTION-ANNEALED MATERIALS

Heat	Specimen Condition °C/h	Test Temperature °C (°F)	0.2% Offset Yield Strength Mpa (ksi)
A	SA(a)	RT(b)	235.8 (34.2)
	538/ 4000	RT	251.0 (36.4)
	593/4000	RT	295.1 (42.8)
	649/4000	RT	283.4 (41.1)
	SA	538 (1000)	105.5 (15.3)
	538/4000	538 (1000)	193.7 (28.1)
	SA	593 (1100)	103.4 (15.0)
	593/4000	593 (1100)	248.2 (36.0)
	SA	649 (1200)	107.6 (15.6)
	649/4000	649 (1200)	247.5 (35.9)
E	SA	RT	192.4 (27.9)
	538/4000	RT	248.2 (36.0)
	593/4000	RT	203.4 (29.5)
	649/4000	RT	185.5 (26.9)
	SA	538 (1000)	123.4 (17.9)
	538/4000	538 (1000)	188.2 (27.3)
	SA	593 (1100)	122.7 (17.8)
	593/4000	593 (1100)	147.5 (21.4)
	SA	649 (1200)	121.4 (17.6)
	649/4000	649 (1200)	115.1 (16.7)

(a) SA: Solution-annealed

(b) RT: Room-temperature

TABLE 3
RESULTS OF X-RAY DIFFRACTION ANALYSIS FOR CARBIDE PHASES FORMED IN THE SPECIMENS OF HEATS A AND E AFTER 500-HR AGING

Aging Temperature °C (°F)	Carbide Precipitates (mg) in 10 g of Alloy					
	Heat A			Heat E		
	$M_{23}C_6$ (mg)	TiN (mg)	Ti(C,N) (mg)	$M_{23}C_6$ (mg)	TiN (mg)	Ti(C,N) (mg)
As-Rec'd	0.6	3	3	5	12	5
538 (1000)	1.4	5	4	8	10	2
593 (1100)	7	6	3	15	9	7
649 (1200)	46	6	6	71	7	11
760 (1400)	40	6	5	100	8	6
816 (1500)	36	4	4	94	9	6

TABLE 4

CHANGES IN 0.2% OFFSET YIELD STRENGTHS [MPa (ksi)] AND AMOUNTS OF CARBIDES FORMED FOR SPECIMENS OF HEATS C AND E AFTER THREE DIFFERENT AGING TREATMENTS

Specimen Condition	Heat E			Heat C		
	Y.S. at RT(a) MPa (ksi)	Y.S. at 593°C(b) MPa (ksi)	Amount of Carbides Formed(c) (mg)	Y.S. at RT(a) MPa (ksi)	Y.S. at 593°C(b) MPa (ksi)	Amount of Carbides Formed(c) (mg)
As-received	192.4 (27.9)	113.8 (16.5)	25	197.9 (28.7)	119.3 (17.3)	19
Aged 593°C (1100°F) 1000 h	191.0 (27.7)	122.0 (17.7)	45	299.2 (43.4)	249.6 (36.2)	52
Aged 760°C (1400°F) 1000 h	182.7 (26.5)	113.8 (16.5)	114	193.7 (28.1)	126.2 (18.3)	79
Aged 760°C (1400°F) 1000 h + 593°C (1100°F) 1000 h	178.6 (25.9)	117.9 (17.1)	101	330.3 (47.9)	282.0 (40.9)	85

(a) 0.2% offset yield strength at room temperature.

(b) 0.2% offset yield strength at 593°C (1100°F).

(c) Amount of carbides ($M_{23}C_6$, Ti(c,n), and TiN) in mg in 10 g alloy.

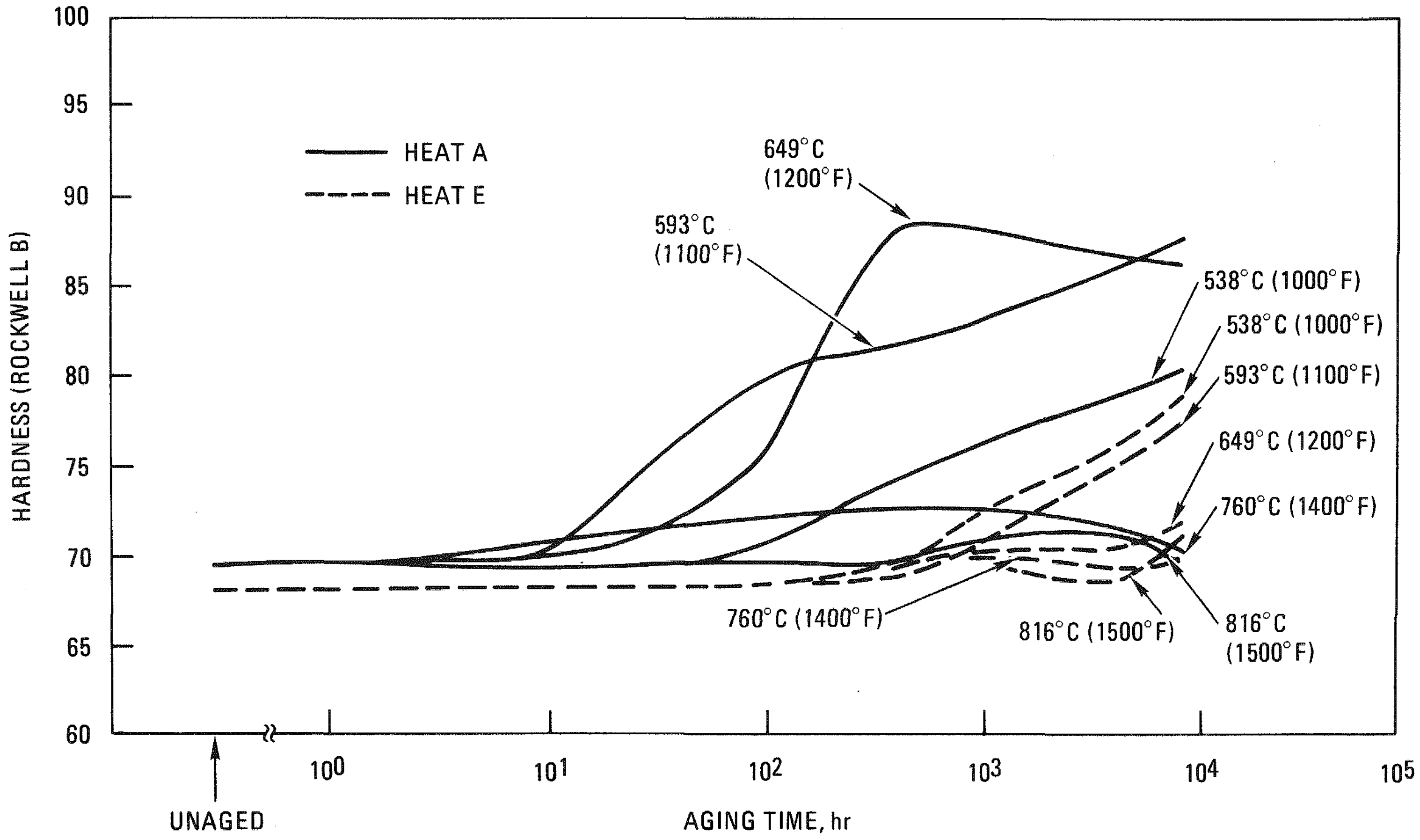

Fig. 1. Comparison of aging behaviors between Heats A and E for times up to 8000 hr

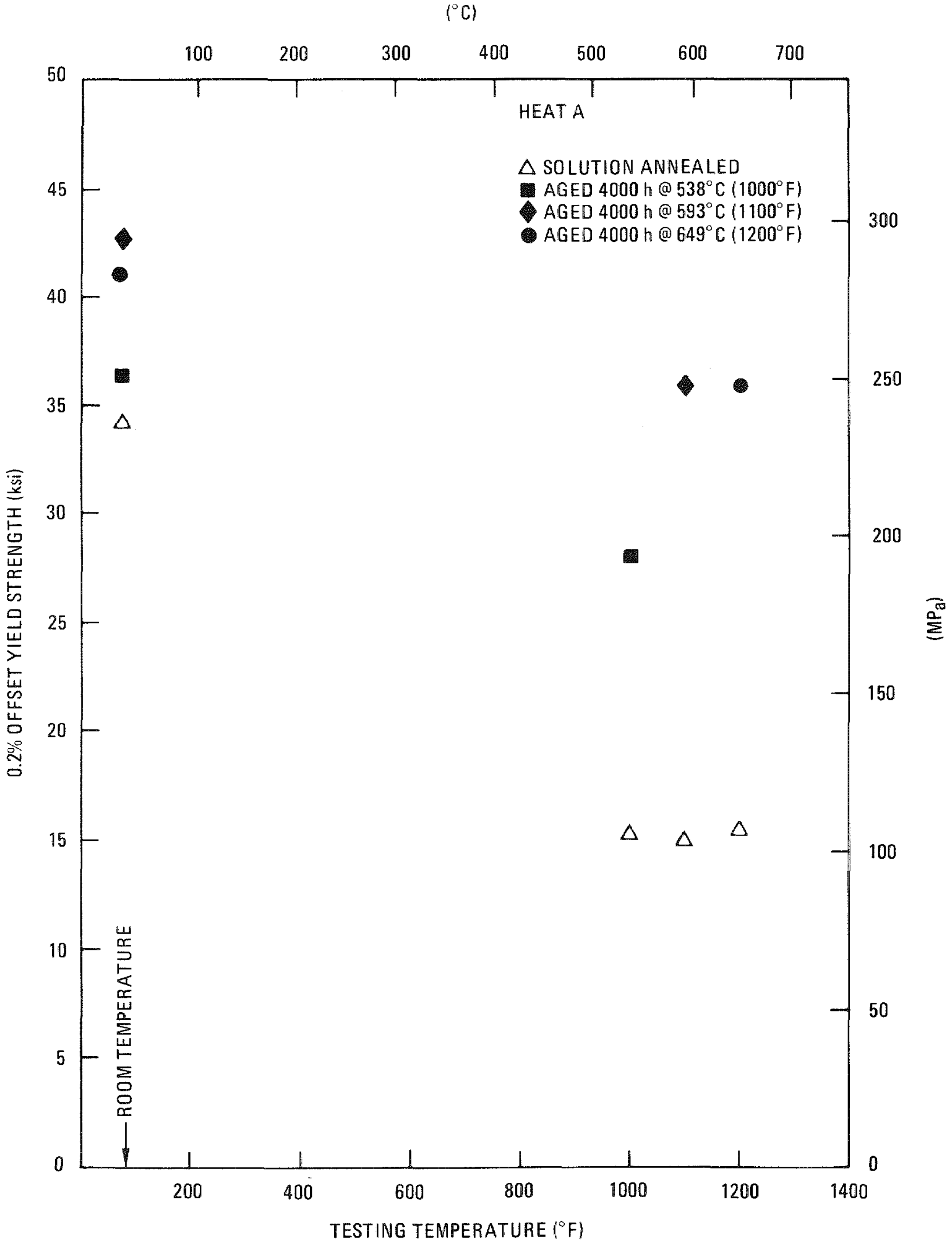

Fig. 2. Comparison of yield strengths between solution-annealed and aged specimens of Heat A.

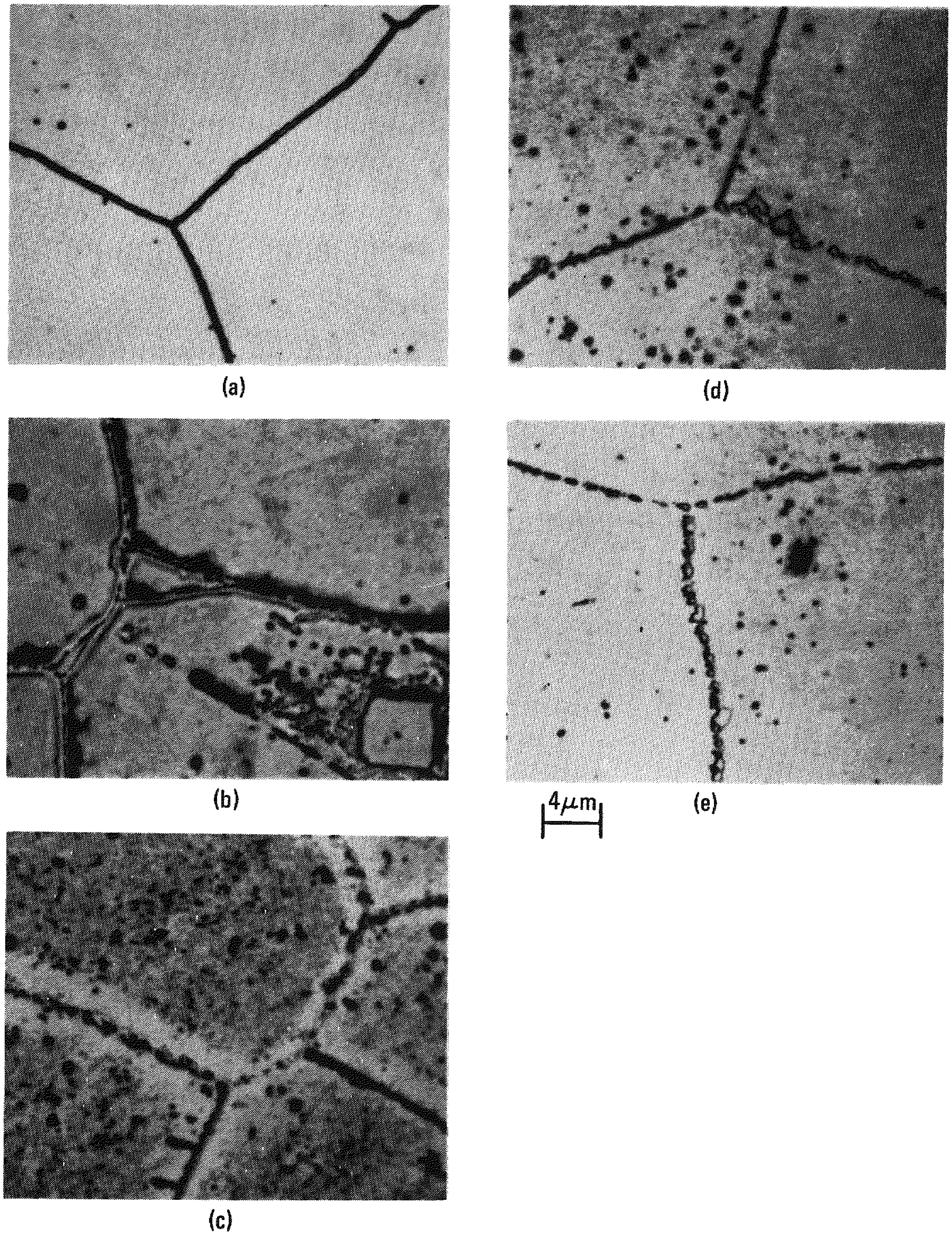

Fig. 3. Optical photomicrographs showing microstructures of specimens of Heat A after 8000-hr aging at (a) 538°C (1000°F), (b) 593°C (1100°F), (c) 649°C (1200°F), (d) 760°C (1400°F), and (e) 816°C (1500°F).

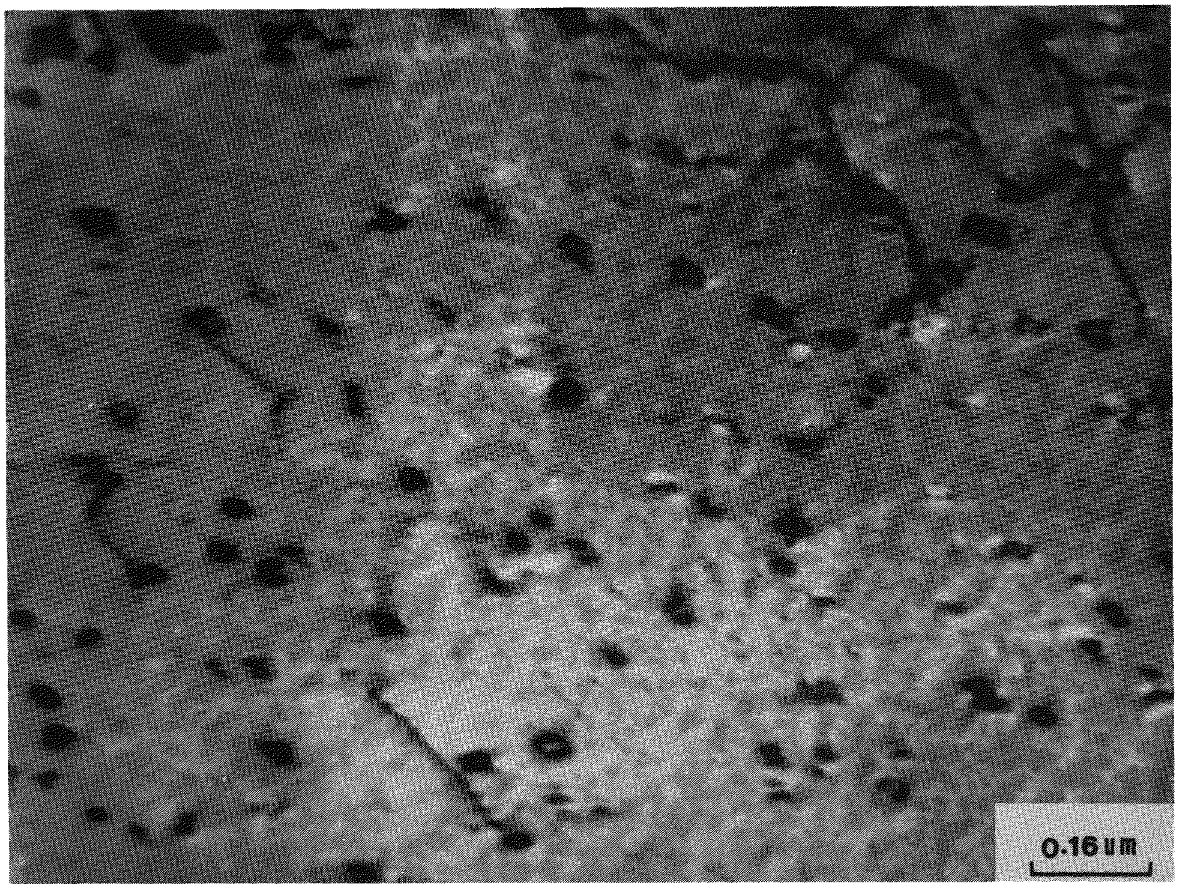

Fig. 4. TEM photomicrograph showing γ' precipitates in the specimen of Heat A aged at 649°C (1200°F) for 96 hrs

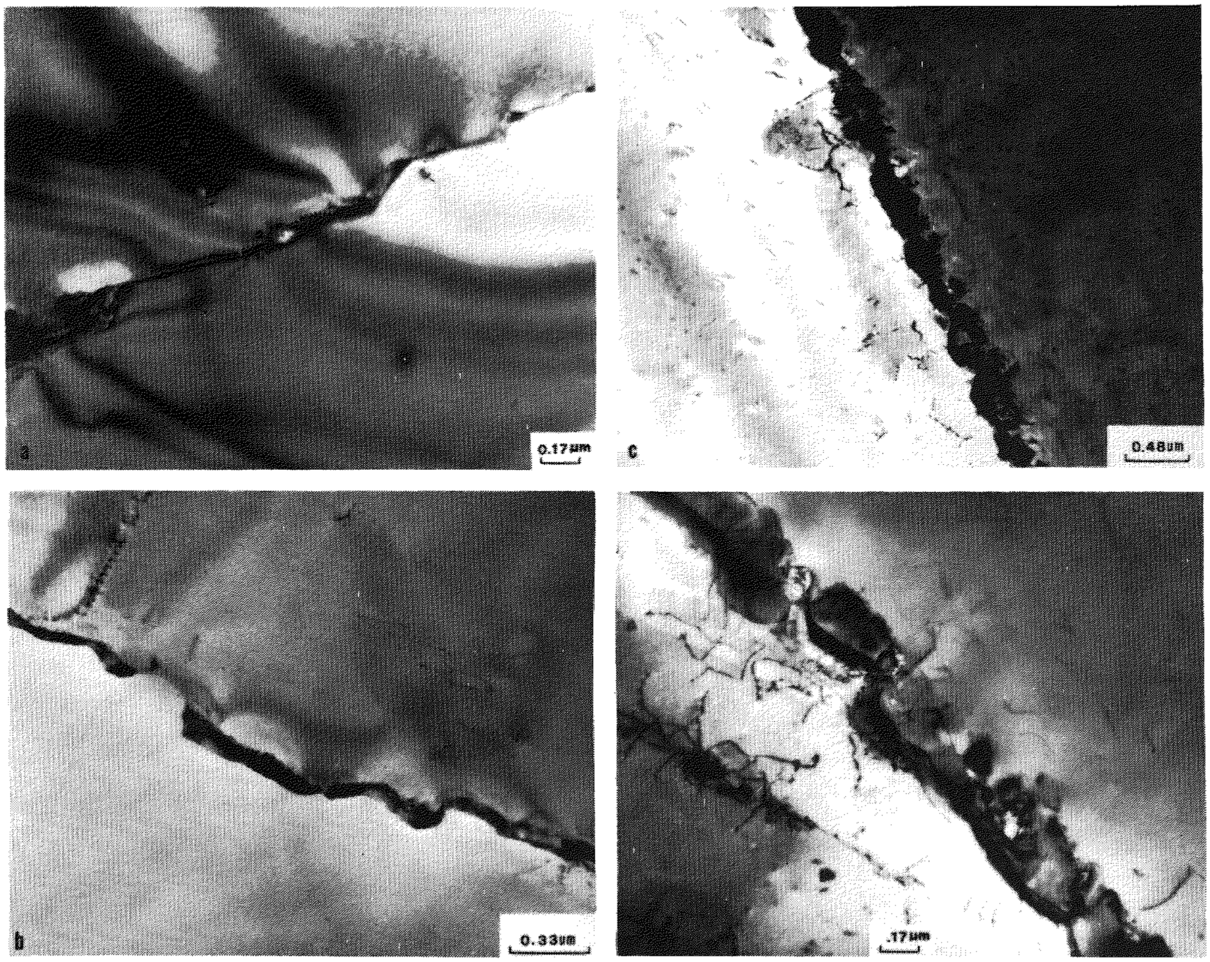

Fig. 5. TEM photomicrographs showing typical grain boundary carbides in the specimens of Heat A after aging for 336 hr at (a) 538°C (1000°F), (b) 593°C (1100°F), (c) 649°C (1200°F) and (d) 760°C (1400°F).

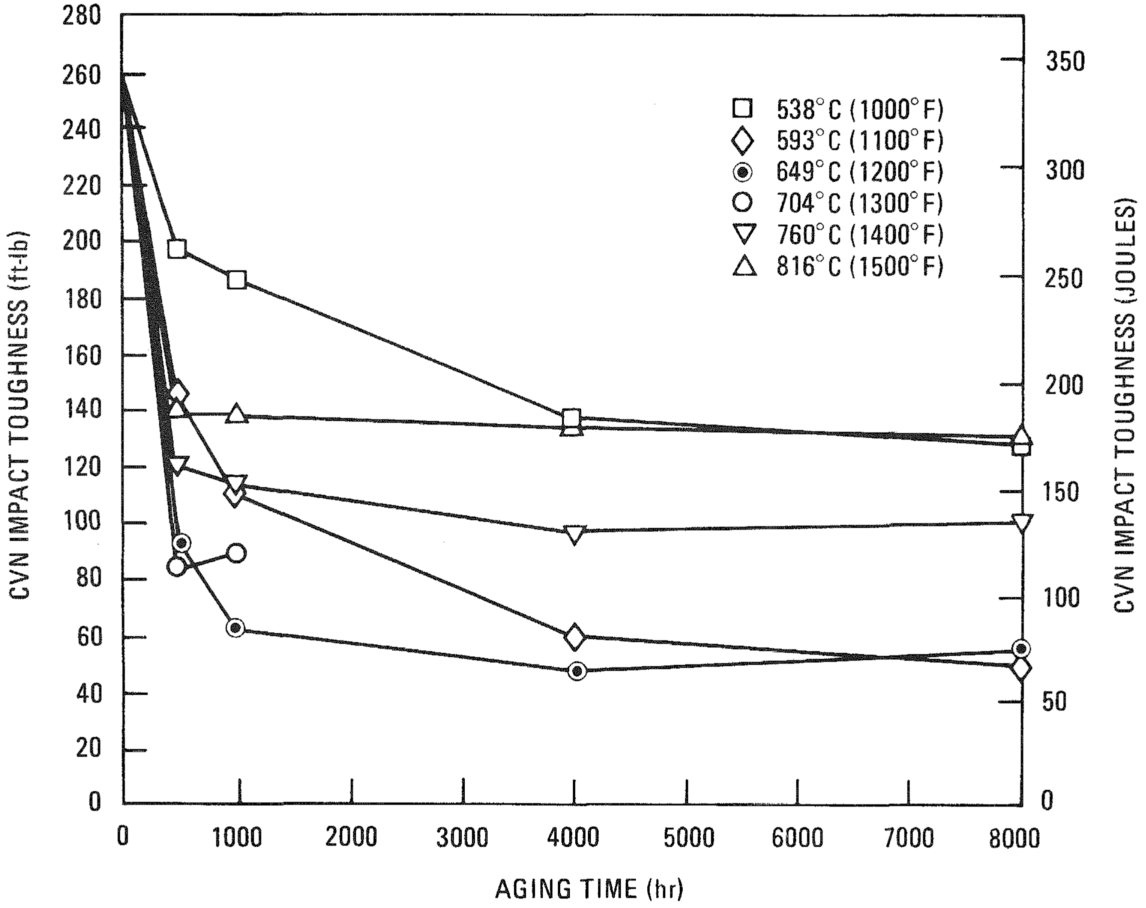

Fig. 6. Changes in room-temperature impact toughness as a function of aging time and temperatures for Heat A

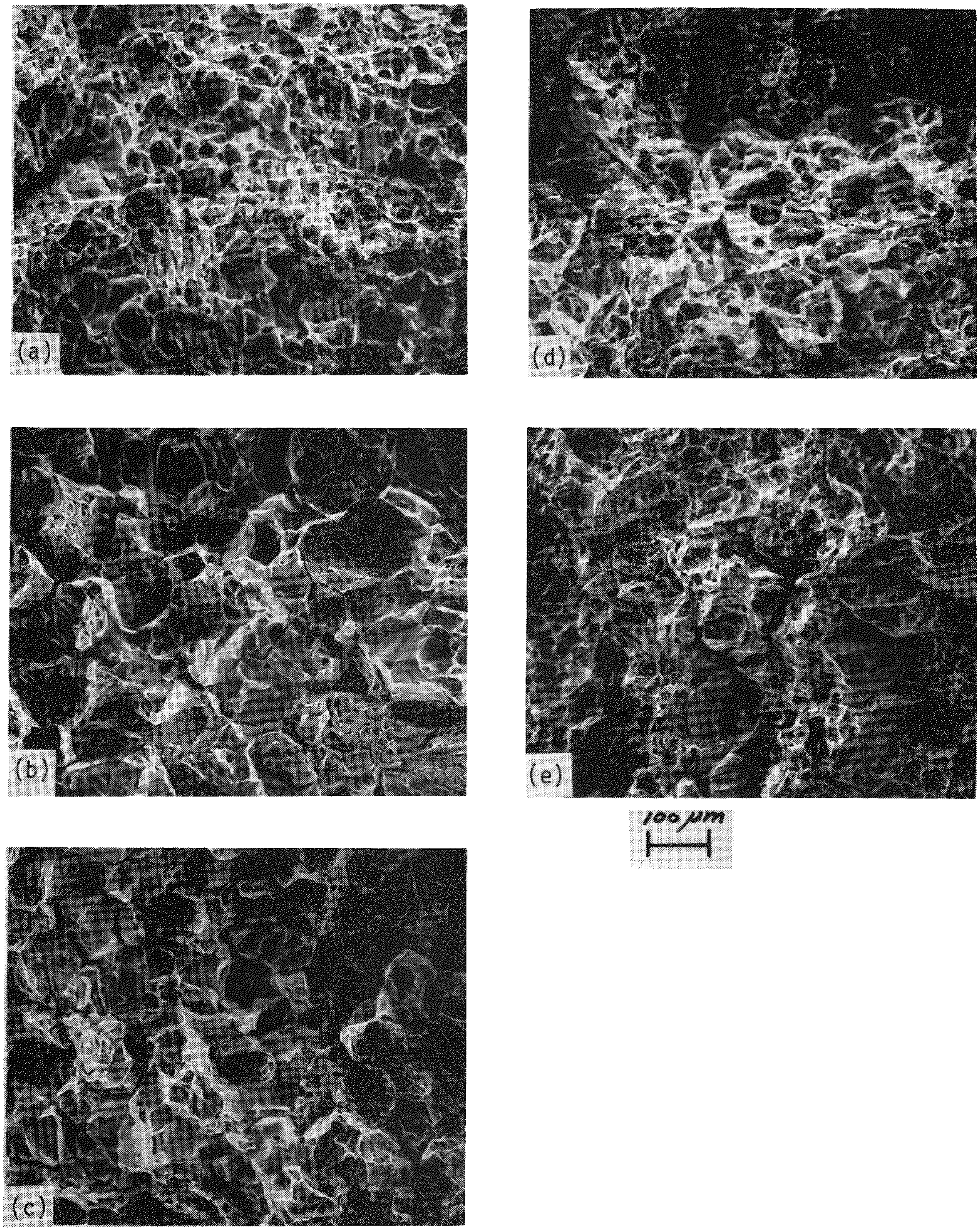

Fig. 7. SEM fractographs showing fracture surfaces of Charpy impact specimens of Heat A aged for 8000 hr at (a) 538°C (1000°F), (b) 593°C (1100°F), (c) 649°C (1200°F), (d) 760°C (1400°F) and (e) 816°C (1500°F).

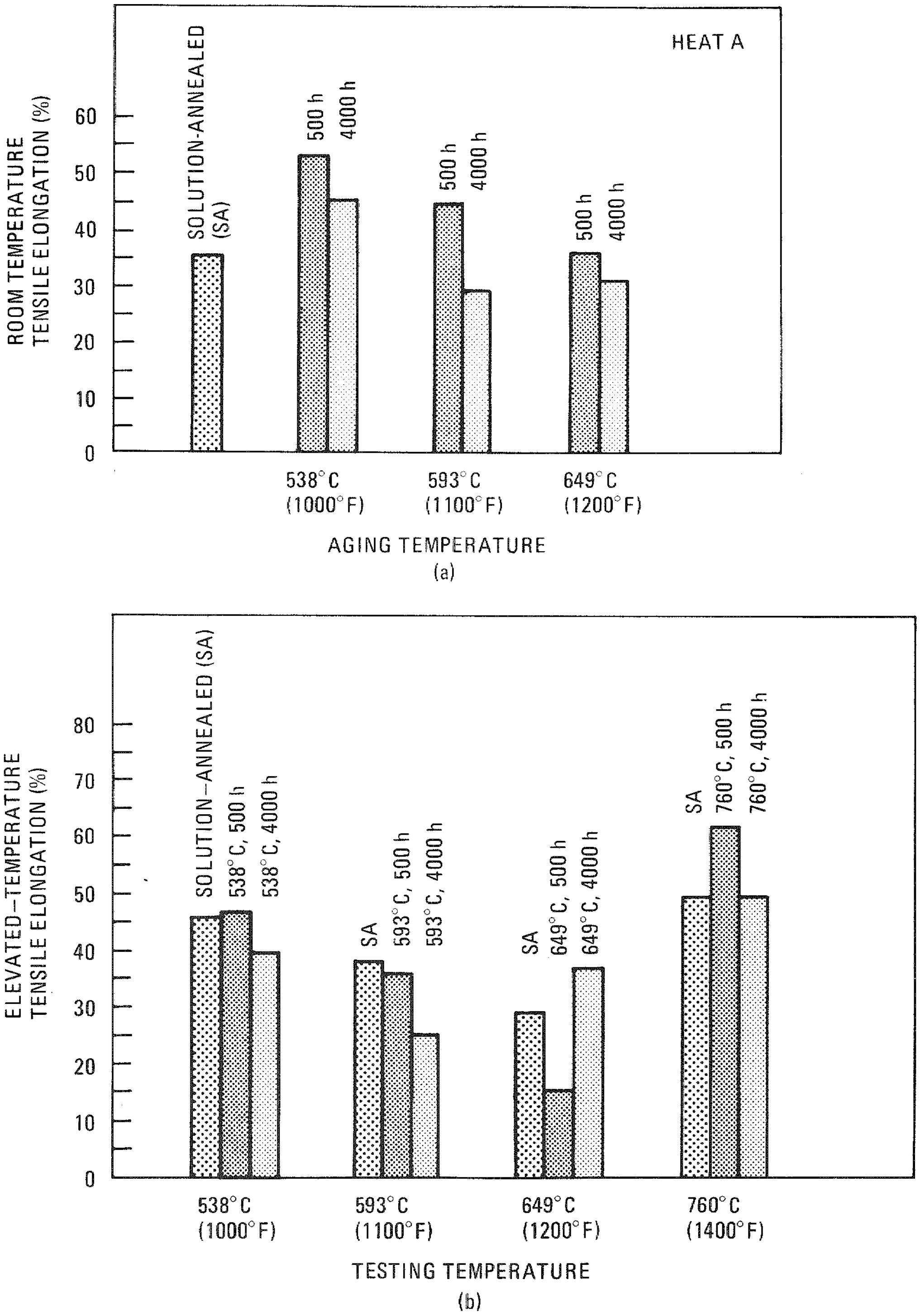

Fig. 8. Changes in room- and elevated-temperature tensile elongation (in 3.81 cm gage length) after thermal aging for Heat A

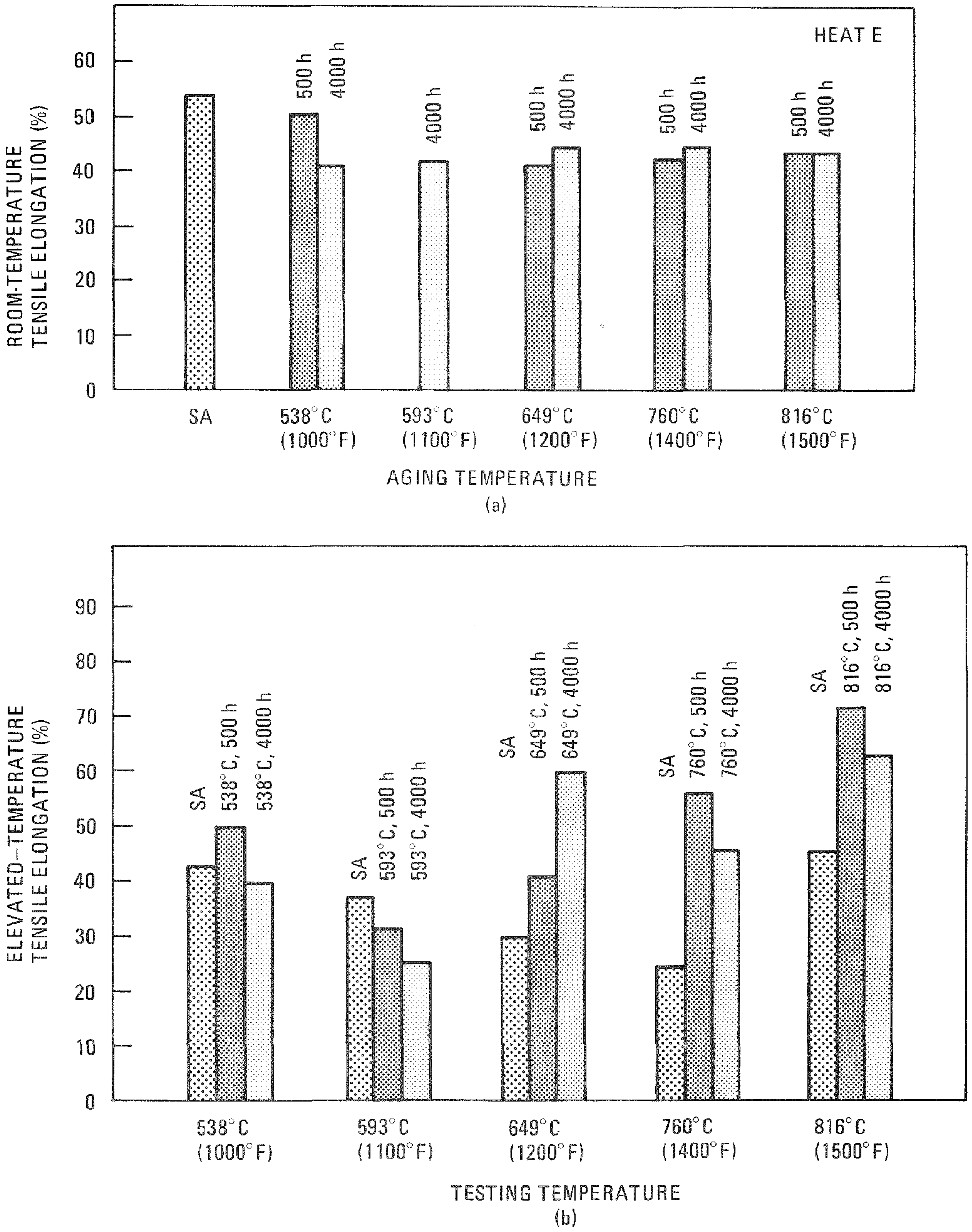

Fig. 9. Changes in room- and elevated-temperature tensile elongation (in 3.81 cm gage length) after thermal aging for Heat E.