ORNL-TM-3734

EFFECT OF AGING TIME AND TEMPERATURE ON THE IMPACT AND TENSILE BEHAVIOR OF L-605 — A COBALT-BASE ALLOY

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OAK RIDGE NATIONAL LABORATORY
OPERATED BY UNION CARBIDE CORPORATION • FOR THE U.S. ATOMIC ENERGY COMMISSION

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ABSTRACT

The effect of aging temperature and time on the impact and tensile behavior of L-605 was studied over the range of 760 to 870°C and 50 to 4000 hr. Prolonged exposure at these temperatures resulted in severe alloy embrittlement. The principal embrittling reaction was the precipitation of the Laves phase, Co_2W. At the lower temperatures, however, the precipitation of Co_3W may have contributed to embrittlement. Maximum aging embrittlement occurred after 500 hr at 870°C.

Impact testing of identically aged specimens at temperatures of 315 to 760°C revealed that 93% of the toughness was lost at the highest test temperature and that all fractures were intergranular. Impact testing at lower temperatures revealed a greater loss of toughness.

Specimens aged at 760, 815, and 870°C and tensile tested at room temperature and 540°C showed a severe loss of ductility, the greatest loss occurring in specimens aged at 870°C. Specimens aged at 760°C for 1000 hr exhibited a ductility of 10% at room temperature and 28% at 540°C. Longer aging times (at 760°C) resulted in a continued loss of ductility.

INTRODUCTION

The Co-Cr-W alloy, L-605 (Haynes alloy No. 25), is generally regarded as one of the most proven cobalt-base alloys commercially available. This alloy is used in many common engineering applications ranging from jet engine components to furnace muffles operating in environments such as air and low-pressure nitrogen or oxygen. In addition, L-605 is used in the construction of liquid metal heat-transfer loops and accessory equipment. The excellent oxidation resistance,[1] corrosion resistance in liquid

[1]Haynes Alloy No. 25 Data Folder, Haynes Stellite Company, Kokomo, Indiana, June 1962. Now Stellite Division, Cabot Corporation, Kokomo, Indiana 46901.

metals,[2] high-temperature strength,[3,4] and good fabricability and weldability promote the use of this alloy in these applications.

The use of L-605 in an environment such as the hard vacuum in space for times in excess of 10,000 hr may, however, lead to serious problems associated with low post-age ductility[5,6] and weld fusion line failures.[7] In addition, resistance to thermal cycle failure could be low because of the loss of low-temperature ductility.

Impact loading of L-605 hardware after long-term aging at 500 to 900°C is possible in some aerospace applications. The present investigation therefore was concerned with the effect of long-term aging on the microstructure, impact, and tensile behavior of L-605.

Alloy L-605 derives its strength primarily from solid-solution hardening, but additional strength can be acquired by aging. Cobalt is hexagonal close packed at room temperature, but changes to face-centered cubic above 418°C. The ductility of a cobalt-base alloy is often affected by this polymorphic transformation of the matrix, with the hexagonal close-packed lattice being associated with the less ductile structures. Tungsten and chromium raise and nickel and iron lower the transformation temperature.[8] For L-605, transformation from face-centered cubic to hexagonal

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[2] D. H. Jansen and E. E. Hoffman, Type 316 Stainless Steel, Inconel, and Haynes Alloy No. 25 Natural-Circulation Boiling-Potassium Corrosion Test Loops, ORNL-3970 (June 1965).

[3] R. Widmer, J. M. Dhosi, and N. J. Grant, Mechanisms Associated with Long Time Creep Phenomena, Technical Report AFML-TR-65-181, Part II, 1967.

[4] C. J. Slunder, Short Term Tensile Properties of the Co–20 Cr–15 W–10 Ni Cobalt-Base Alloy (L-605), DMIC Memo No. 179, Battelle Memorial Institute (Sept. 27, 1963).

[5] E. E. Jenkins, Embrittlement of Haynes Alloy No. 25 During Brazing, Report No. 817-1390, Haynes Stellite Company, Kokomo, Indiana (May 21, 1958).

[6] S. T. Wlodek, Embrittlement of a Co-Cr-W (L-605) Alloy, R-61-FPD-538 (Dec. 1, 1961).

[7] R. W. Swindeman, Oak Ridge National Laboratory, private communication, February 1969.

[8] F. S. Badger and F. C. Kraft, Jr., "Cobalt-Base and Nickel-Base Alloys for Ultrahigh Temperature," Metal Progr. 52, 394 (1947).

close packed can be expected between 650 and 900°C, but only after very long times.[9,10] This sluggishness is a prerequisite for retention of high-temperature strength over long periods of time.

EXPERIMENTAL PROCEDURE

The purchasing of materials, machining of the test specimens, and all heat treatments in air were completed at the Martin-Marietta Corporation, the Martin Company, Baltimore, Maryland. The impact testing was conducted at the Battelle Memorial Institute, Columbus, Ohio, while the tensile testing was completed at the Martin Company. Evaluation of all data, metallographic examination, hardness and grain size determinations, chemical analysis, and final reporting were completed at ORNL. Additional impact specimens were machined, aged in argon, and tested at ORNL for the purpose of determining environmental effects and the aging response of L-605 at lower temperatures.

Materials

Two pieces of L-605 were purchased from the UCC Stellite Division in accordance with aerospace material specification AMS-5537B. The chemical composition, mechanical properties (vendor's), and heat identifications are given in Table 1. The material as received had been solution heat-treated (in air) at 1230°C for a period of not less than 0.25 hr.

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[9] W. Koster, "On the Influence of the Elements on the Polymorphic Transformation of Cobalt," Z. Metallk. 43, 297 (1952).

[10] A. R. Elsea and C. C. McBride, "The Effects of Nitrogen, Iron, and Nickel on the Alpha/Beta Transformation and Gamma Precipitation in Cobalt-Chromium Alloys," Trans. AIME 188, 154 (1950).

Table 1. Analyses of Commercial L-605

Composition[a] (wt %)	Mechanical Properties	Heat Number 186-6-1843 (0.056 in. sheet)	Heat Number 186-5-1737 (0.500 in. plate)
Cr		20.24	20.37
W		15.02	14.81
Fe		2.00	1.80
Ni		10.24	10.00
Mn		1.35	1.43
C		0.09	0.11
Si		0.03	0.18
S		0.005	0.014
P		0.007	0.016
Co		Balance	Balance
	Ultimate tensile strength, psi	136,650	129,800
	Yield strength 0.2% offset, psi	64,750	59,200
	Ductility, in 2 in.	56.5%	64%

[a]Vendor's analysis.

Specimen Machining and Heat Treatment

The Charpy V-notch impact specimens and sheet tensile specimens were machined in accordance with Fig. 1. All specimens were subsequently solution annealed at 1230°C for a period of not less than 0.25 hr. The impact specimens were aged in air at 870°C for periods of 50, 100, 500, 1000, 2500, and 4000 hr. The sheet tensile specimens were aged in air at 760, 820, and 870°C for periods of 50, 100, 500, 1000, and 4000 hr. Additional impact specimens were machined, solution annealed at 1230°C for 1 hr in vacuum, and aged in argon at 650, 760, and 870°C for 50, 200, 500, and 1000 hr. These additional specimens were machined from the same heat (186-5-1737) as were the air-aged specimens.

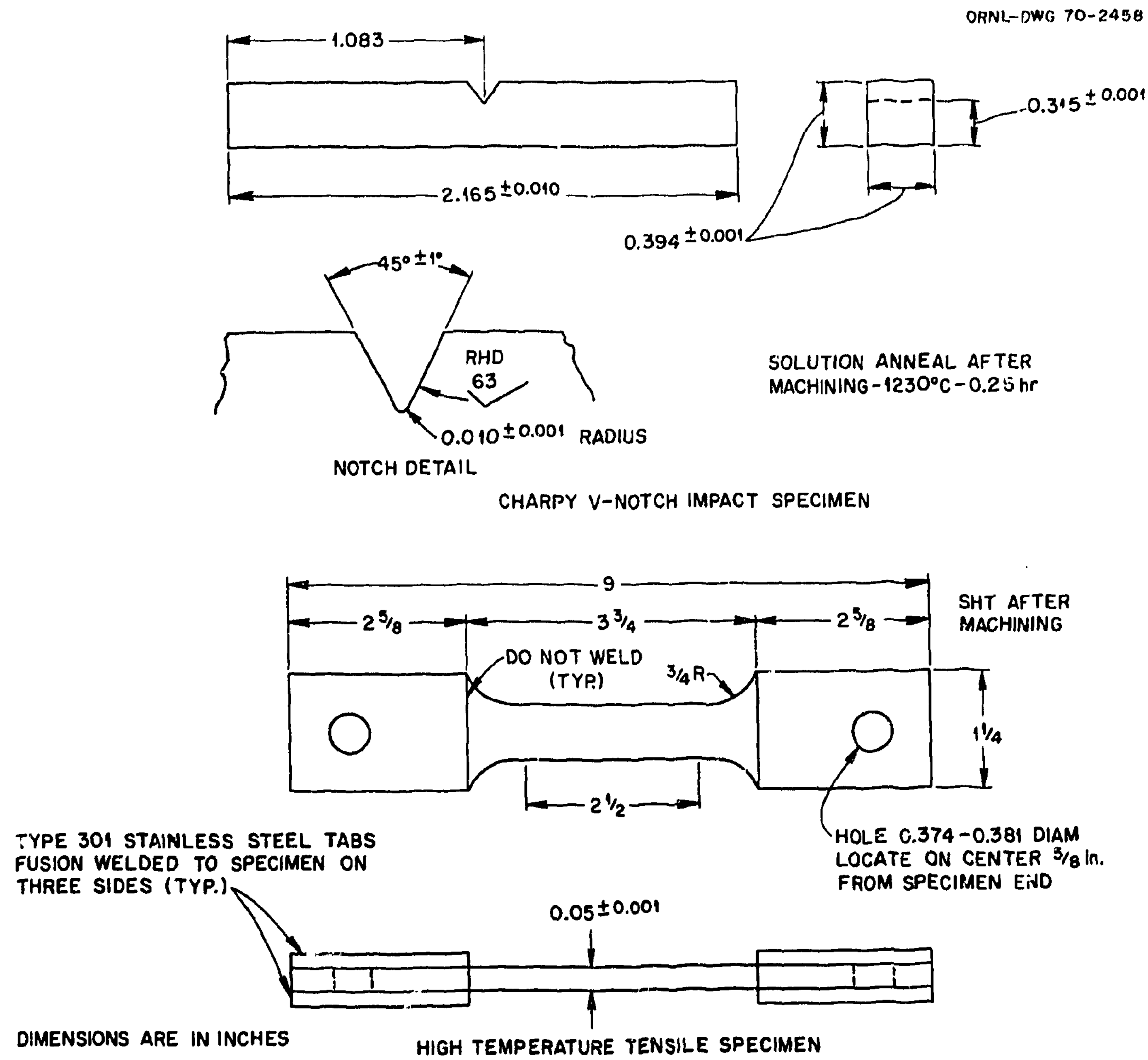

Fig. 1. Specimen Specifications for L-605 Aging Study.

Impact Testing

A certified Richle impact tester (serial No. RA-17876) was used to test the specimens aged in air. Specimens were impact tested in air at 315, 427, 538, 649, and 760°C. A transfer time from the furnace to the testing block of 4 to 5 sec necessitated a temperature calibration of the specimen to correct for resulting thermal losses. A small hole was machined in a spare specimen to permit a Chromel-P–Alumel thermocouple (28-gage wire) to be located directly behind the notch. The thermocouple connected to a high-speed recorder showed that specimens impact tested at the above temperatures had to be heated to 317, 430, 543, 656, and 770°C, respectively. The instrumented specimen was heated with the specimen to be tested as a more accurate indicator of specimen temperature. Two specimens of each aging treatment were impact tested at the same temperature.

In an effort to simulate the heating by reentry into the earth's atmosphere, impact specimens aged in air for 4000 hr at 870°C were heated to 1110°C and tested at approximately 1080°C.

The impact specimens solution annealed in vacuum and aged in argon were impact tested at 316°C with a Baldwin tester (serial No. 1075). The specimens were superheated 2 to 3°C. Spare specimens aged in air were impact tested on the Baldwin machine; the results were identical to those obtained on the Richle tester at the Battelle Memorial Institute.

Tensile Testing

A certified Baldwin Lima Hamilton Universal tensile testing machine was used in making tensile measurements of air-aged specimens. The tensile tester was equipped with a pacing device for the measurement and control of the strain rate at 0.05 in./hr. Three specimens of each aged condition were tensile tested at 540°C, and one specimen of each aged condition was tested at room temperature. In addition, three specimens aged for 4000 hr at 870°C were tensile tested at 1080°C in an effort to simulate reentry heating.

DISCUSSION OF RESULTS

Impact Tests

Initially, it should be understood that the uninstrumented Charpy V-notch impact test possesses many shortcomings. It does not provide numerical information that can be used directly in design calculations, the stress distribution during fracture is unknown, the resistance of the material to crack initiation and propagation cannot be obtained, and conditions of the test do not adequately reflect actual service conditions. In this study the (notched) impact test was used to determine relative changes in the toughness of L-605 microstructures that resulted from thermal treatments which will be encountered in service and which are known to cause alloy embrittlement. These results coupled with calculations of reentry velocities and impact forces associated with reentry and launch pad aborts will hopefully determine the serviceability of L-605 under impact loads. In addition, the notched specimen geometry may simulate notches of the type encountered in welds and regions of high stress concentration.

The Charpy V-notch impact test is a severe mechanical test. An alloy withstanding a Charpy test without exhibiting much low energy cleavage has an excellent chance of withstanding a cleavage fracture in service at the test temperature. When the maximum fracture toughness is greater than nominally 100 ft-lb (at 100% tear), it is considered sufficiently high to guarantee against failure by low energy tear, at least in medium-strength materials. Conversely, when the fracture toughness is low (5 to 20 ft-lb) and the fracture is principally by cleavage or is intergranular, the alloy is very susceptible to brittle failure.

Aged at 870°C

The fracture toughness of L-605 aged in air at 870°C generally decreased with aging time at all test temperatures. The greatest decrease occurred after aging for 50 hr. A recovery in toughness was observed after the 100-hr aging treatment, but thereafter the toughness continued to decrease for aging times to 4000 hr. This behavior is illustrated in Fig. 2. For specimens impact tested at 427°C, a 100% fibrous fracture was obtained for the solution-annealed condition, while subsequent aging treatments resulted in slight cleavage, gross intergranular fractures, and a small increase in grain size. The 50-hr aging treatments, for example, resulted in about 20% cleavage (transgranular tear without deformation), while after the 100-hr aging treatment, cleavage fractures were very scarce and intergranular fractures were obtained. Aging times longer than 100 hr resulted in total intergranular failures. Hardness behavior (Fig. 3) and lateral expansion further indicate alloy embrittlement and loss of toughness with aging time.

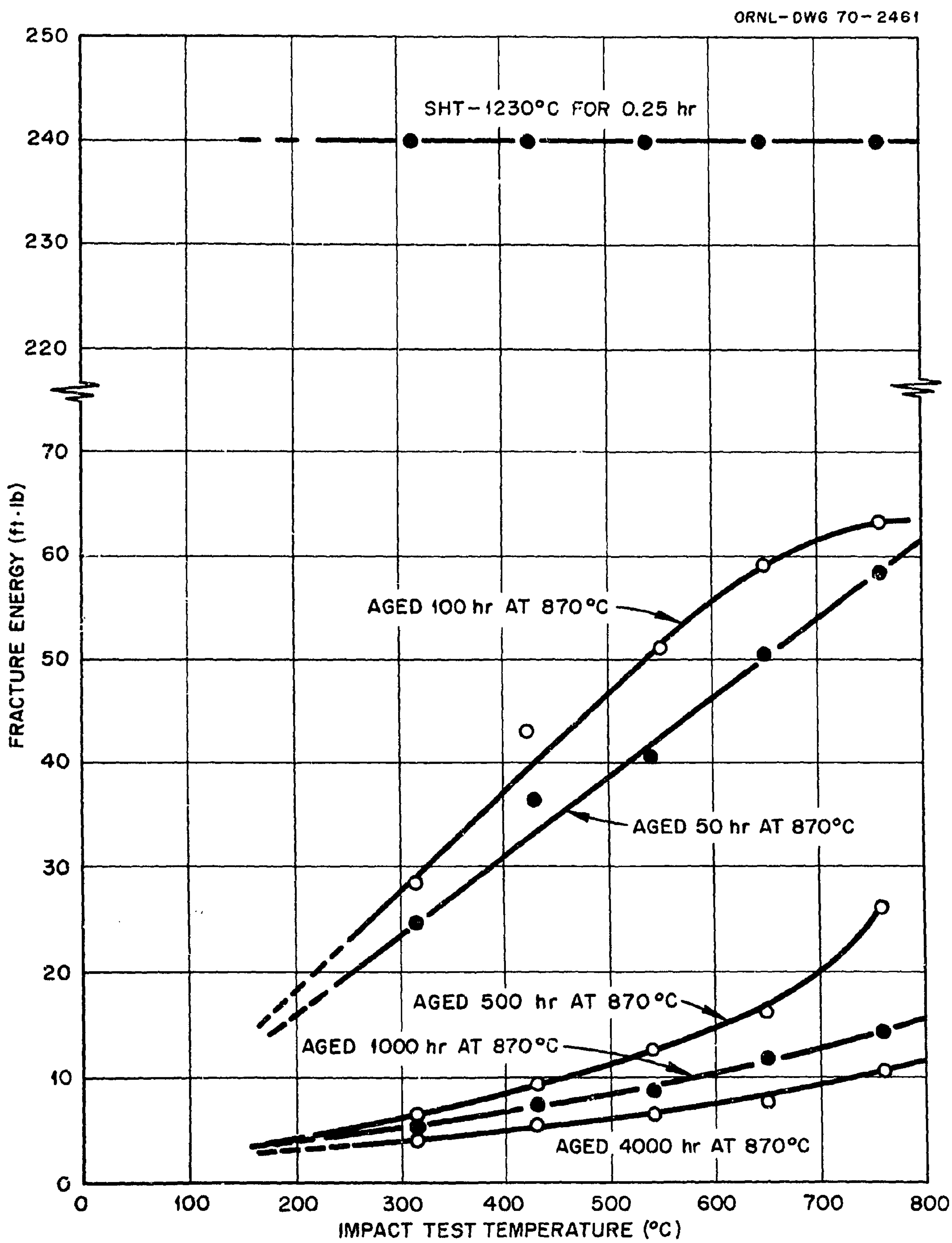

Fig. 2. Influence of Test Temperature and Aging Time in Air at 870°C on the Fracture Toughness of L-605.

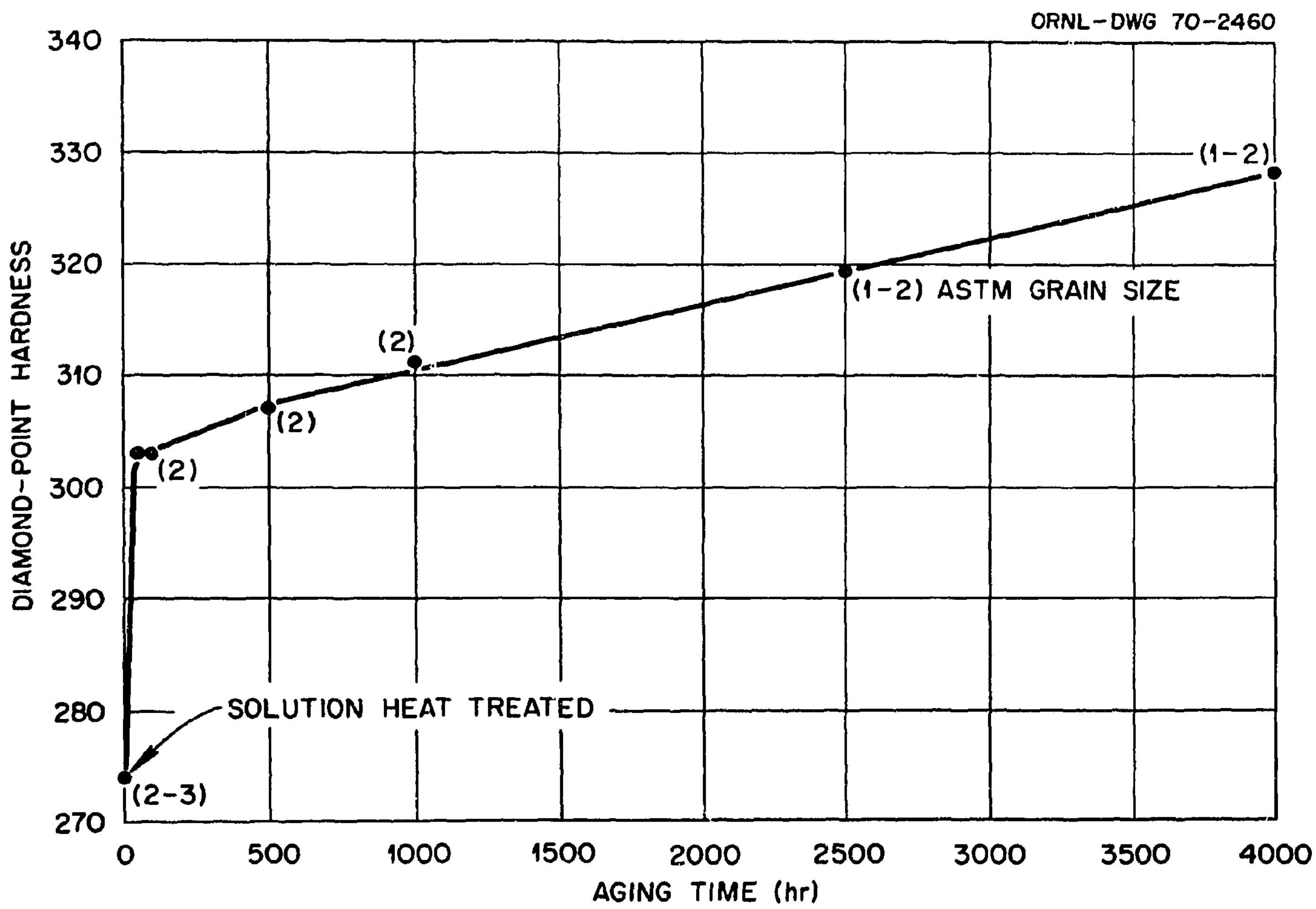

Fig. 3. Effect of Aging Time at 870°C on the Hardness of L-605.

Typical microstructural changes are presented in Fig. 4. The solution-annealed microstructure is essentially a single-phase matrix with scattered heterogeneous carbide dispersions, probably the M_6C type. Transgranular tear with considerable deformation was observed. This condition is typical of a material with high fracture toughness.

Specimens aged at 870°C for 50 hr fractured intergranularly with considerable deformation; however, slight transgranular tear was also observed. This behavior is attributed in part to the precipitation of an irregular, blocky carbide in the grain matrices and an almost continuous spheroidal carbide in the grain boundaries, as shown in Fig. 5. The results of Yukawa and Sato[11] (at 700 and 800°C) indicate that the

[11]N. Yukawa and K. Sato, "The correlation Between Microstructure and Stress Rupture Properties of a Co-Cr-Ni-W (HS-25) Alloy," Trans. Japan Inst. Metals 9, (Supplement) (1968).

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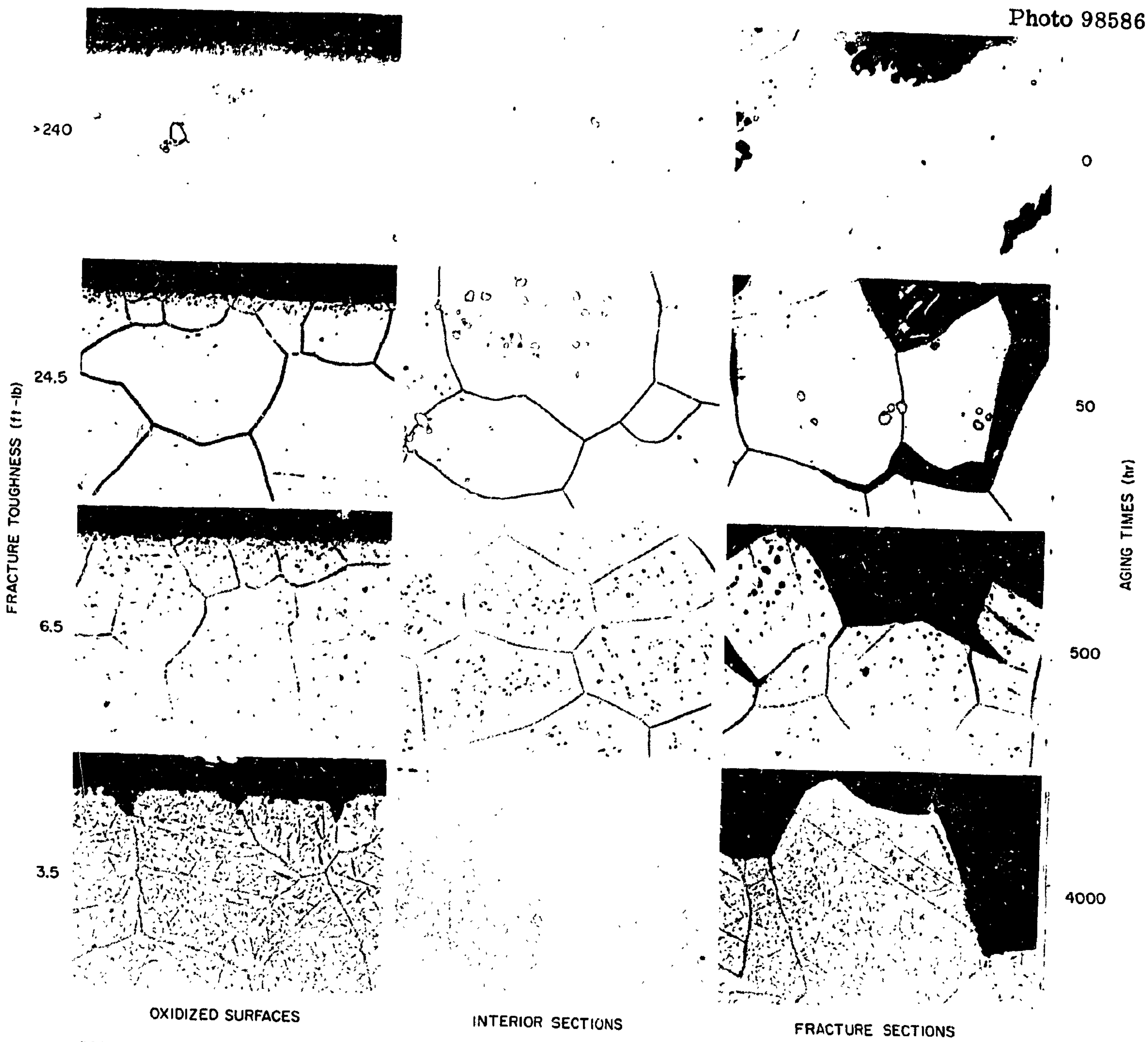

Fig. 4. Microstructural Changes in L-605 Aged in Air at 870°C and Impact Tested at 315°C. Etchant: 100 ml H_2O + 40 ml CH_3COOH + 40 ml HCl + 15 ml H_2SO_4 + 25 g $FeCl_3$. 500X. Reduced 16.5%.

Fig. 5. L-605 Aged for 50 hr at 870°C in Air and Impact Tested at 315°C. Etchant: 100 ml H_2O + 40 ml CH_3COOH + 40 ml HCl + 15 ml H_2SO_4 + 25 g $FeCl_3$. 2000X.

blocky carbide is of the $M_{23}C_6$ type, which initially precipitates in the grain boundaries and subsequently within the grains. More important, however, is that the present studies, conducted at a slightly greater temperature (870°C), have shown that a cobalt-tungsten Laves phase also precipitated (discontinuously) near the grain boundaries during the 50-hr aging treatment and in part is responsible for the intergranular failures and loss of toughness. These results are illustrated in Figs. 5 and 6. The microstructure illustrating the 50-hr age in Fig. 6 shows the initial precipitation of the Laves phase adjacent to the grain boundaries.

Aging for 100 hr resulted in further dissolution or transformation of the grain boundary spheroidal carbide to a finer carbide dispersion

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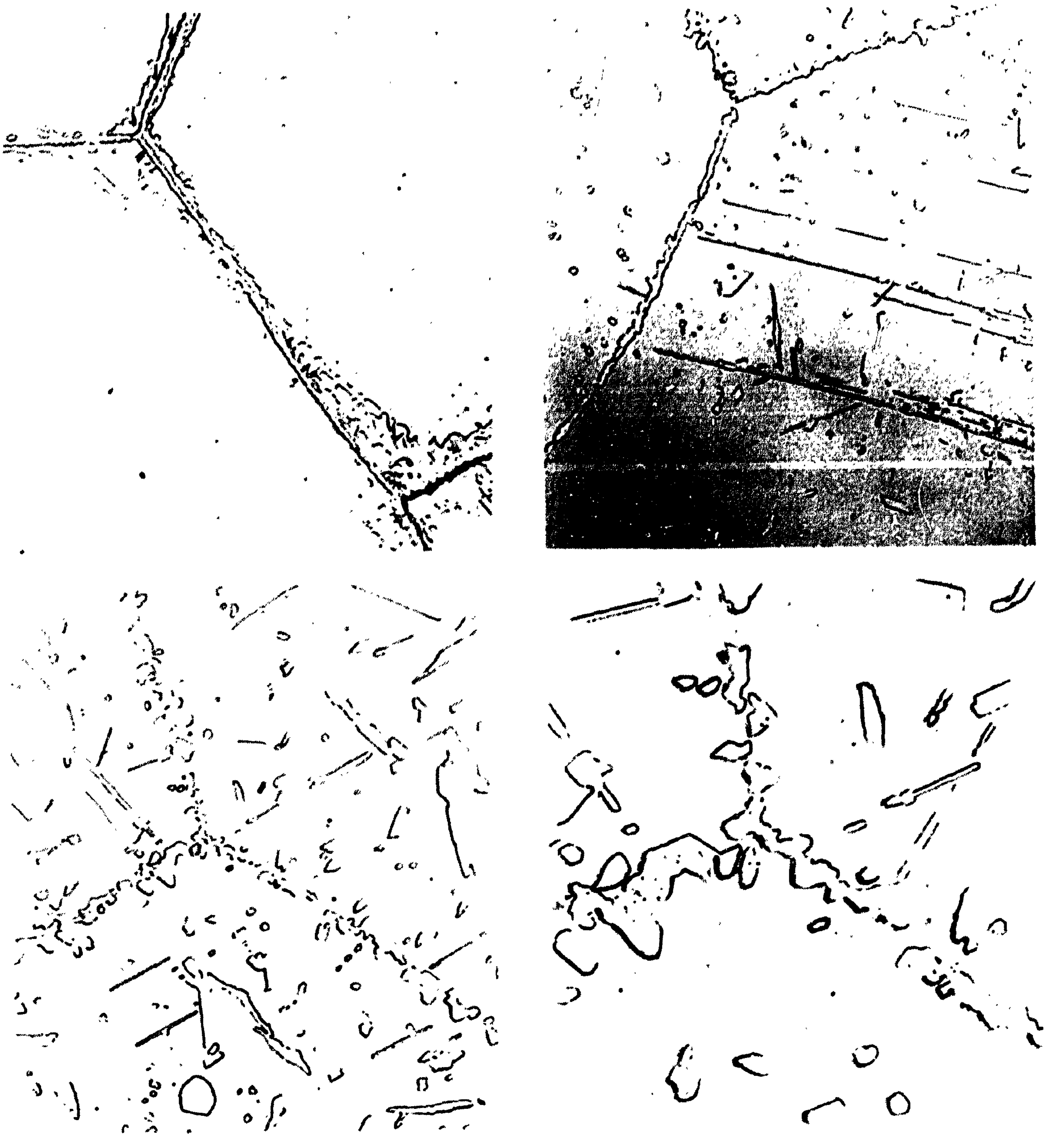

Fig. 6. Electron Micrographs Illustrating the Aging Behavior of L-605 at 870°C. (a) Aged 50 hr, 5000×. (b) Aged 500 hr, 5000×. (c) Aged 4000 hr, 5000×. (d) Aged 4000 hr, 12,500×.

in the matrix. Grain boundaries and adjacent areas, however, contained an increased amount of Laves phase. Yukawa,[12] Jenkins,[13] and Wlodek[14] showed that blocky $M_{23}C_6$ transforms to M_6C and that after 100 hr at temperatures above 800°C, the Laves phase, $(Co,Ni)_2(Cr,W)$, precipitates near the grain boundaries as elongated platelets and subsequently as intergranular particles. The results of the present study are in agreement with the results of the previous investigations. For simplicity, the Laves phase will be designated Co_2W.

Continued aging for times to 4000 hr at 870°C merely resulted in a fine dispersion of the M_6C carbide and an increased amount of Co_2W, which precipitated in the grain boundaries, on twin boundaries, and probably on stacking faults. At an intermediate aging time of 500 hr, the behavior is illustrated in Fig. 6. The Laves phase thickened and coarsened with time and depleted the matrix of some components. For example, chemical analysis showed that the tungsten concentration in the matrix decreased from 14.81 to 7.46 wt % during 1000 hr at 870°C. The drastic loss of fracture toughness and apparent loss of ductility are attributed principally to the precipitation of the Laves phase in the grain boundaries.

Aged at 760°C

The fracture toughness resulting from aging in argon at 760°C is shown in Fig. 7 for an impact test temperature of 316°C. The greatest loss of fracture toughness due to aging at 760°C again occurred during the initial 50 hr. The 100-hr age, however, did not result in a recovery of fracture toughness. Instead, the toughness gradually decreased with aging time at a rate less than for specimens aged at 870°C. This behavior is attributed to the slower precipitation rate of the embrittling Laves phase at 760°C. Metallographic examination supported the contention that

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[12] N. Yukawa and K. Sato, "The Correlation Between Microstructure and Stress Rupture Properties of a Co-Cr-Ni-W (HS-25) Alloy," Trans. Japan Inst. Metals 9, (Supplement) (1968).

[13] E. E. Jenkins, Embrittlement of Haynes Alloy No. 25 During Brazing, Report No. 817-1390, Haynes Stellite Company, Kokomo, Indiana, (May 21, 1958).

[14] S. T. Wlodek, Embrittlement of a Co-Cr-W (L-605) Alloy, R-61-FPD-538 (Dec. 1, 1961).

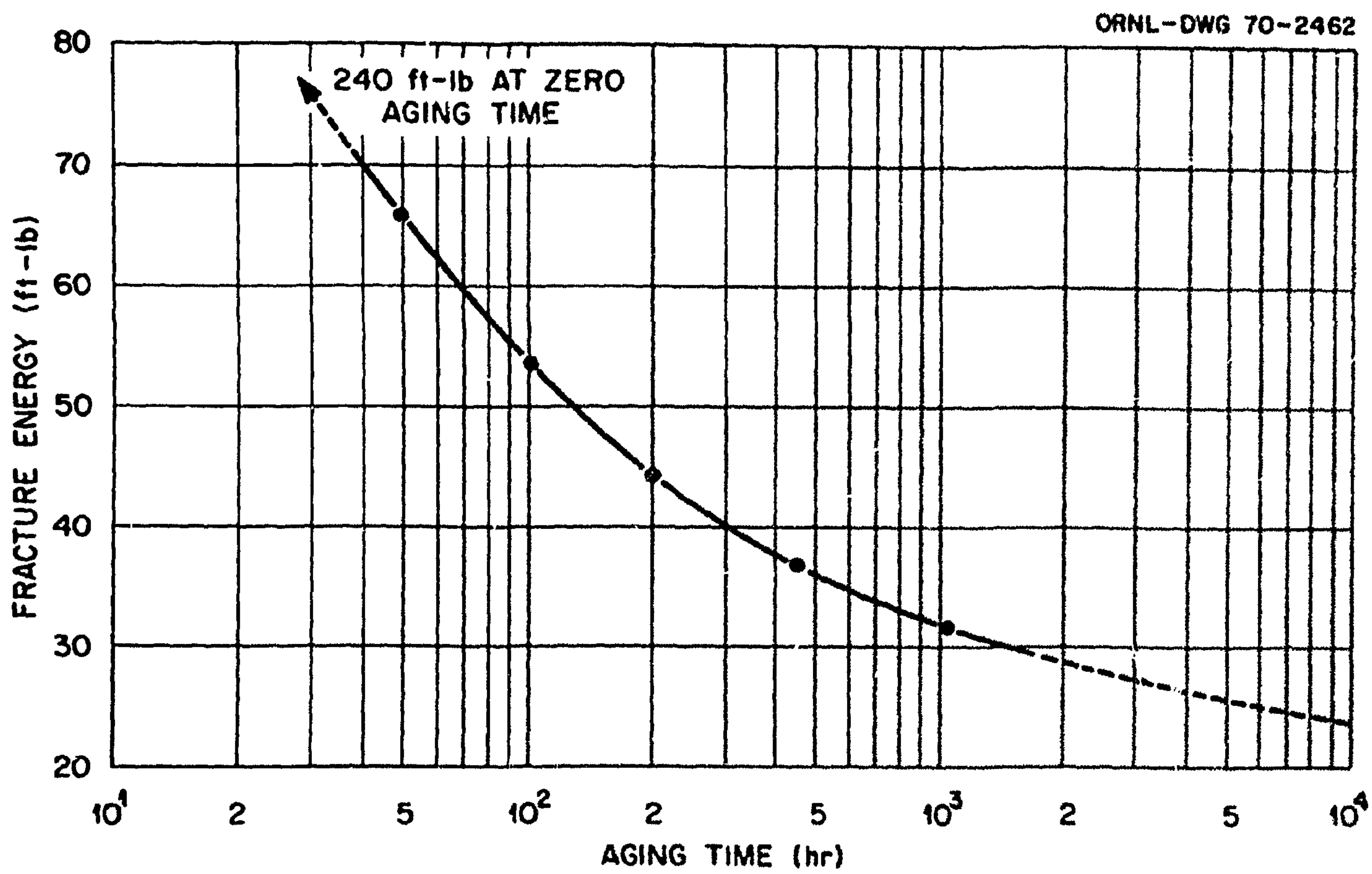

Fig. 7. Effect of Time on the Fracture Toughness of L-605 Aged in Argon at 760°C and Tested at 316°C.

there was vastly less Laves phase in the grain boundaries of specimens aged at 760°C for equal periods and that for times to 500 hr considerably more cleavage failure was present. A comparison of Fig. 8 with Fig. 4 will illustrate these observations. In addition, it is seen that aging in air oxidizes the surface of L-605 principally at the grain boundaries to depths that increase with time; however, these effects are not considered serious in terms of impact resistance.

A shortage of specimens prevented aging treatments longer than 1000 hr at 760°C. However, the necessity of knowing longer term behavior resulted in the extrapolation shown in Fig. 7. It is apparent that the fracture toughness decreased at a rapid rate, and that specimens aged for 448 hr (Fig. 8) exhibited total intergranular fracture. It was concluded, therefore, that L-605 aged for greater periods than 1000 hr at 760°C would be brittle, be sensitive to impact loading, and fracture at impact energies between 20 and 30 ft-lb.

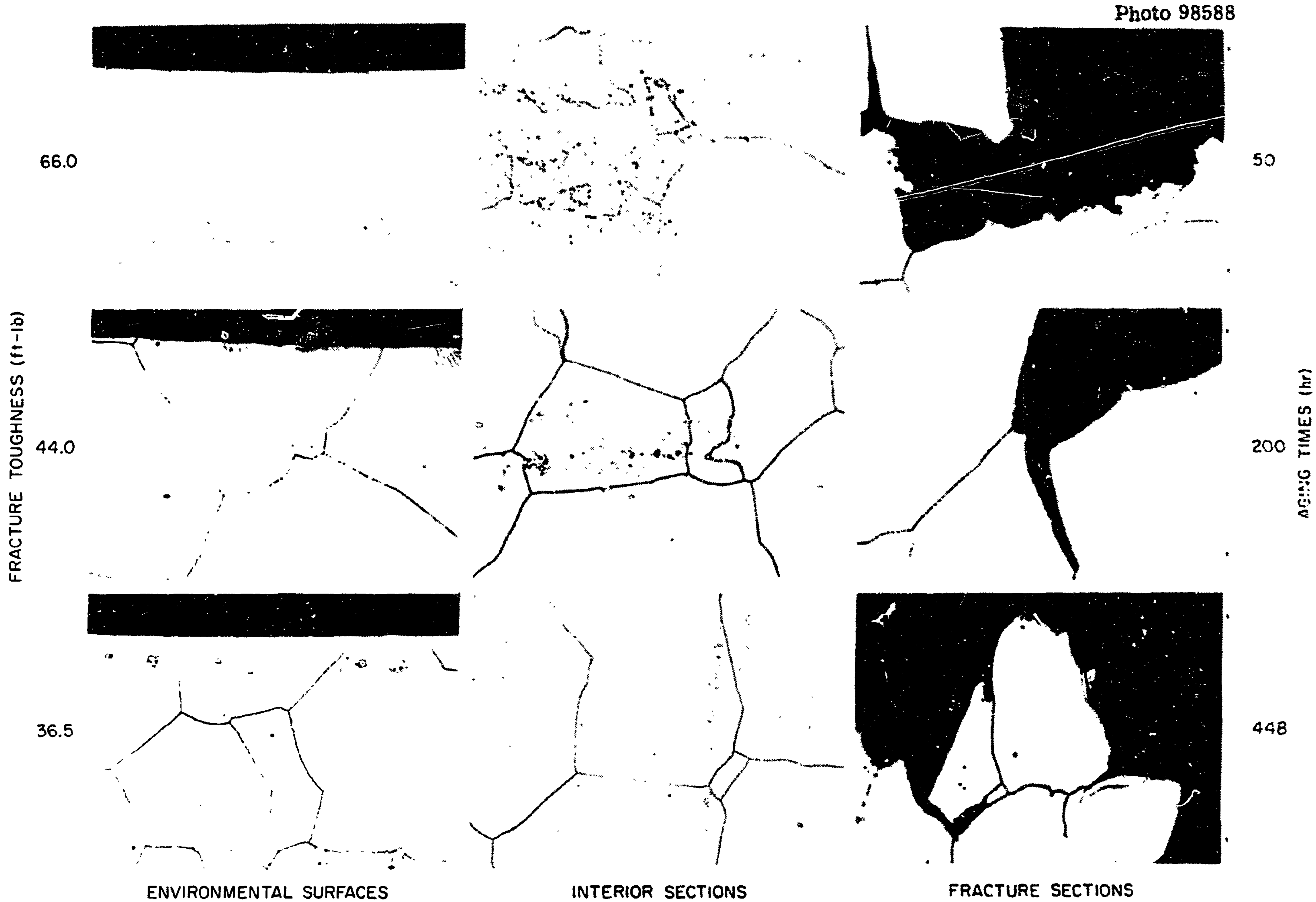

Fig. 8. Microstructural Changes in L-605 Aged in Argon at 760°C and Impact Tested at 316°C. Etchant: 100 ml H_2O + 40 ml CH_3COOH + 40 ml HCl + 15 ml H_2SO_4 + 25 g $FeCl_3$. 500×. Reduced 10.5%.

Aged below 760°C

The effect of aging temperature on the fracture toughness of L-605 is shown in Fig. 9. At temperatures generally above 700°C, aging in air or an inert atmosphere for 500 hr or longer will cause embrittlement. At aging temperatures of 650°C or lower, the fracture toughness (240 ft-lb) for an aging time of 448 hr is the same as for the solution-annealed condition. The time-temperature-transformation diagram derived by Yukawa and Sato[12] indicates that thousands of hours are necessary for embrittling phase to precipitate at these low temperatures. The fracture mode of specimens aged at the lower temperatures is transgranular (high energy) tear with considerable deformation. A typical high-energy tear fracture shown in Fig. 10 was obtained after aging at 650°C and testing at 316°C.

Reentry Heating Effects

Reentry heating caused by the earth's atmosphere was simulated by heating aged specimens to 1110°C for 200 sec. For example, specimens aged for 4000 hr at 870°C were impact tested at 1080°C. Nine specimens showed fracture toughnesses in the range 18.0 to 20.5 ft-lb, and one held at 1110°C for 400 sec showed 21.0 ft-lb. These values are about what would be expected by extrapolation of the curve for 4000 hr aging in Fig. 2 (p. 9). Thus, reentry heating times of 200 to 400 sec are apparently not sufficiently long for toughness recovery.

Tensile Tests

The strain rates associated with normal tensile tests are many times less than those encountered during impact loading. However, the conclusions regarding the embrittlement of L-605 due to aging are the same as those obtained from the impact tests. The L-605 alloy, over the wide range of aging conditions studied, retained sufficient tensile strength and ductility for many conventional (tensile) applications at temperatures above 540°C.

The effect of aging time and temperature on the tensile behavior of L-605 is illustrated in Figs. 11 and 12. The increase in yield and ultimate tensile strength occurring during the initial 500 hr of aging at

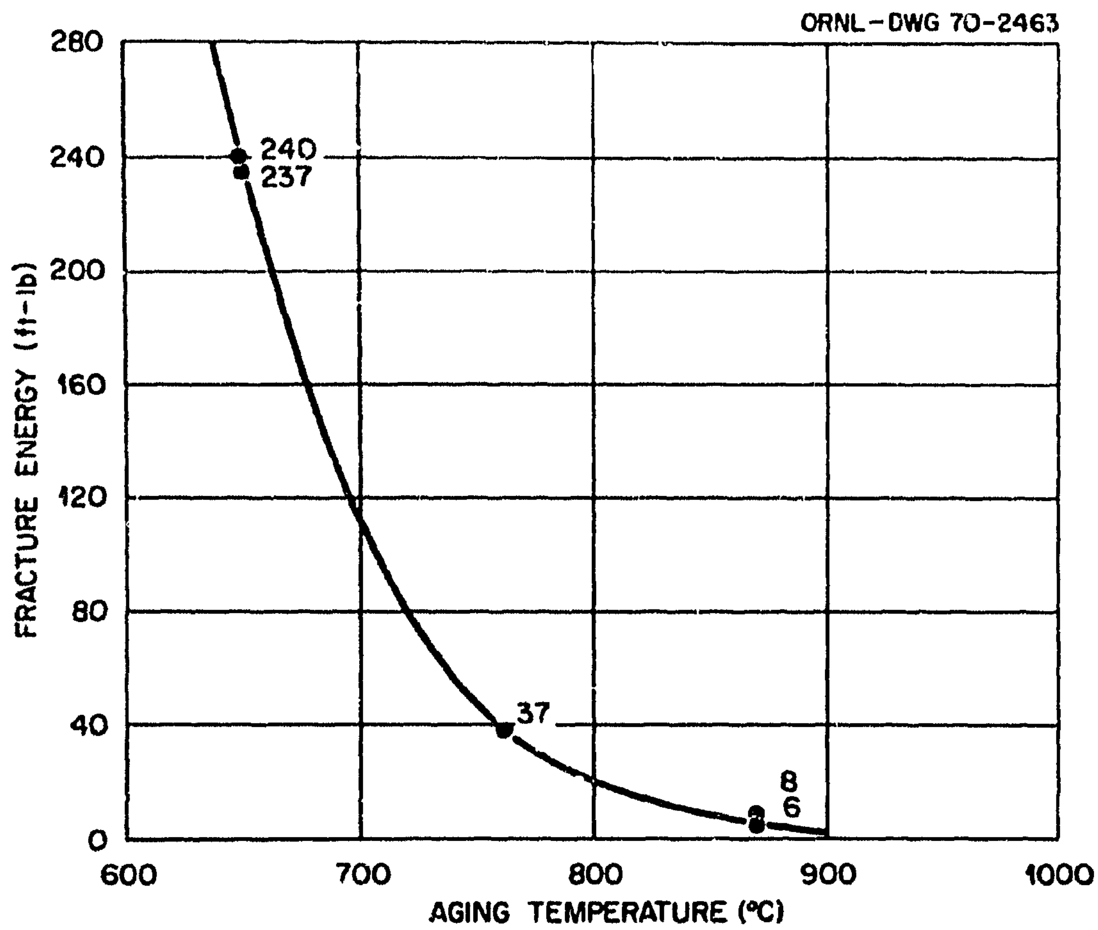

Fig. 9. Effect of Aging Temperature on the Fracture Toughness of L-6 5 Aged in Argon for 448 hr and Tested at 316°C.

Fig. 10. Fracture Mode of L-605 Aged in Argon at 650°C for 448 hr and Impact Tested at 316°C. Etchant: 100 ml H_2O + 40 ml CH_3COOH + 40 ml HCl + 15 ml H_2SO_4 + 40 ml HNO_3 + 25 g $FeCl_3$, electropolished. 500×. Reduced 15%.

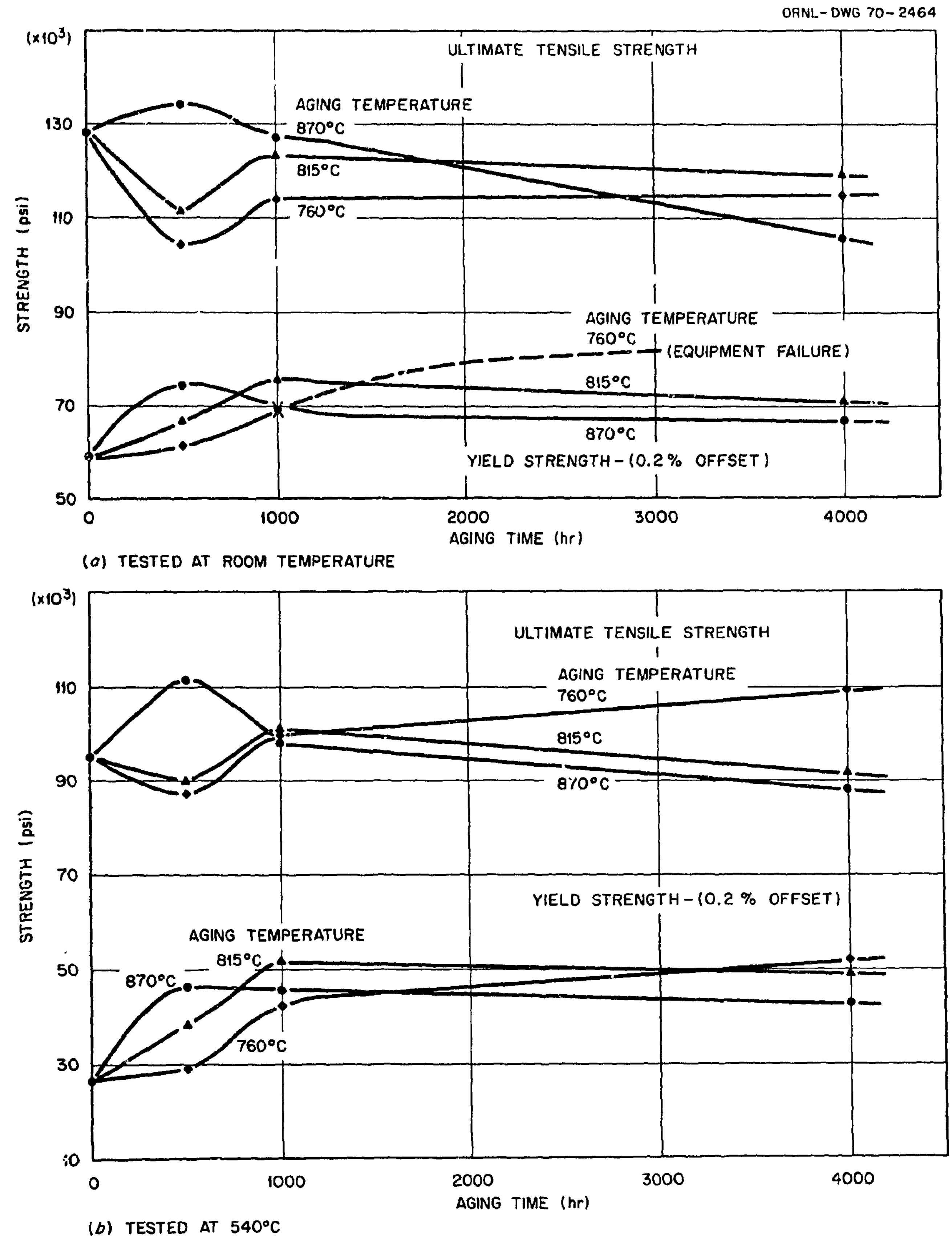

Fig. 11. The Effect of Aging Time and Temperature on the Tensile Properties of L-605 Tested at Room Temperature and at 540°C.

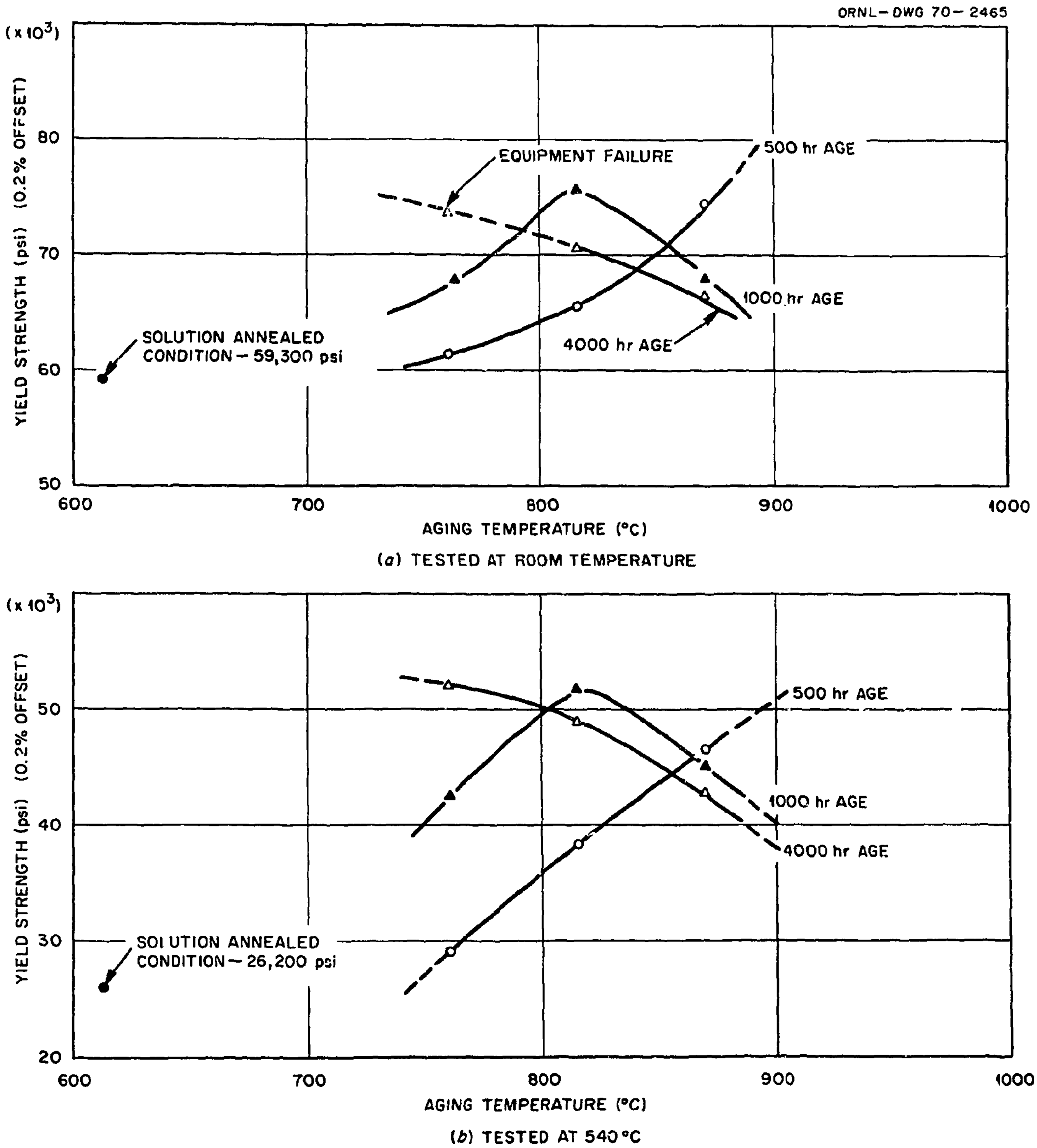

Fig. 12. The Effect of Aging Temperature on the Yield Strength (0.2% Offset) of L-605 Tested at Room Temperature and at 540°C.

870°C is attributed to precipitation of carbides ($M_{23}C_6$ and M_6C) and Co_2W. Between 500 and 4000 hr the Laves phase (Co_2W) coarsened and the quantity increased (overaging) in both the grain boundaries and matrices, resulting in a decrease in the yield and ultimate tensile strengths at both room temperature and 540°C. This loss of strength is attributed in part to the depletion of tungsten from the matrix. Continued precipitation and growth of both the Laves phase (Co_2W) and carbides ($M_{23}C_6$ and M_6C) also resulted in severe loss of tensile ductility, as illustrated in Fig. 13.

The ultimate tensile strength decreased during the initial 500 hr as a result of aging at 815 and 760°C (Fig. 11). However, the yield strength (0.2% offset) increased slightly with a loss of tensile ductility (Fig. 13). Aging for 1000 hr at 760 and 815°C resulted in an increase in both yield and ultimate tensile strengths and a further loss of ductility. The following is offered as an explanation for this behavior.

Aging at 760°C for 500 hr promoted precipitation of $\underline{M_7C_3}$, $M_{23}C_6$, M_6C, $\underline{\alpha\text{-}Co_3W}$, β-Co_3W, and Co_2W (ref. 15), where the underlined phases are metastable. Initially the α-Co_3W phase is coherent with the matrix, but after long aging times it transforms to the noncoherent β-Co_3W phase.[15] Aging times longer than 1000 hr at 760°C resulted in greater ultimate tensile and yield strengths because at this low temperature the aging reactions are still incomplete. Microstructures illustrating the effect of aging time at 760°C are shown in Fig. 14. Increased aging time merely resulted in greater precipitation and embrittlement, as exhibited by the intergranular fractures. Aging at 815°C will probably result in the same behavior as exhibited at 760°C but at greater rates.

At 870°C not only do the aging reactions and hence embrittlement occur at greater rates (than at 815 and 760°C), but only $M_{23}C_6$, M_6C, and Co_2W are found in the microstructure.[15–17] The microstructures of

[15] N. Yukawa and K. Sato, "The Correlation Between Microstructure and Stress Rupture Properties of a Co-Cr-Ni-W (HS-25) Alloy," Trans. Japan Inst. Metals 9, (Supplement) (1968).

[16] E. E. Jenkins, Embrittlement of Haynes Alloy No. 25 During Brazing, Report No. 817-1390, Haynes Stellite Company, Kokomo, Indiana (May 21, 1958).

[17] S. T. Wlodek, Embrittlement of a Co-Cr-W (L-605) Alloy, R-61-FPD-538 (Dec. 1, 1961).

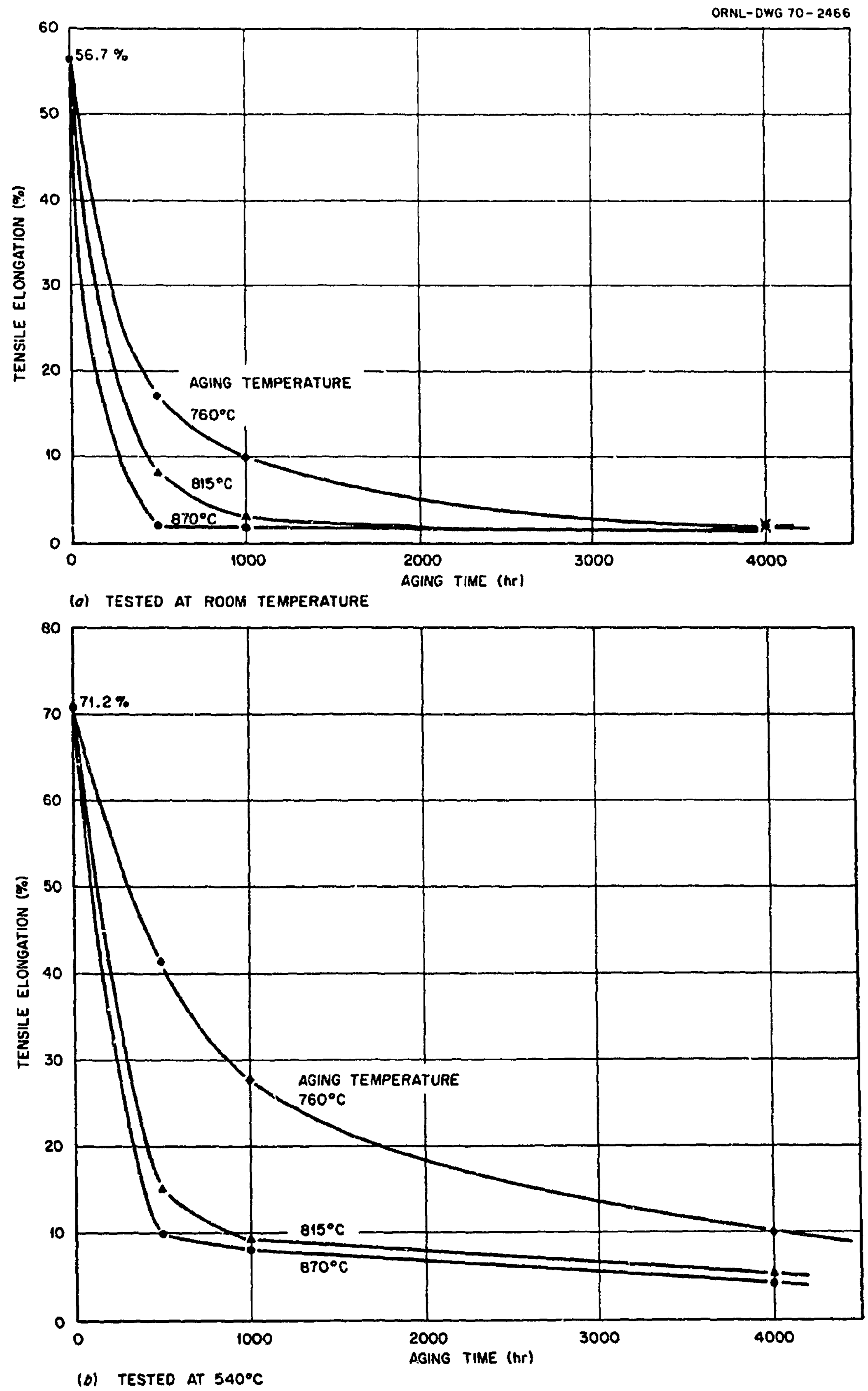

Fig. 13. The Effect of Aging Time and Temperature on the Tensile Ductility of L-605 Tested at Room Temperature and at 540°C.

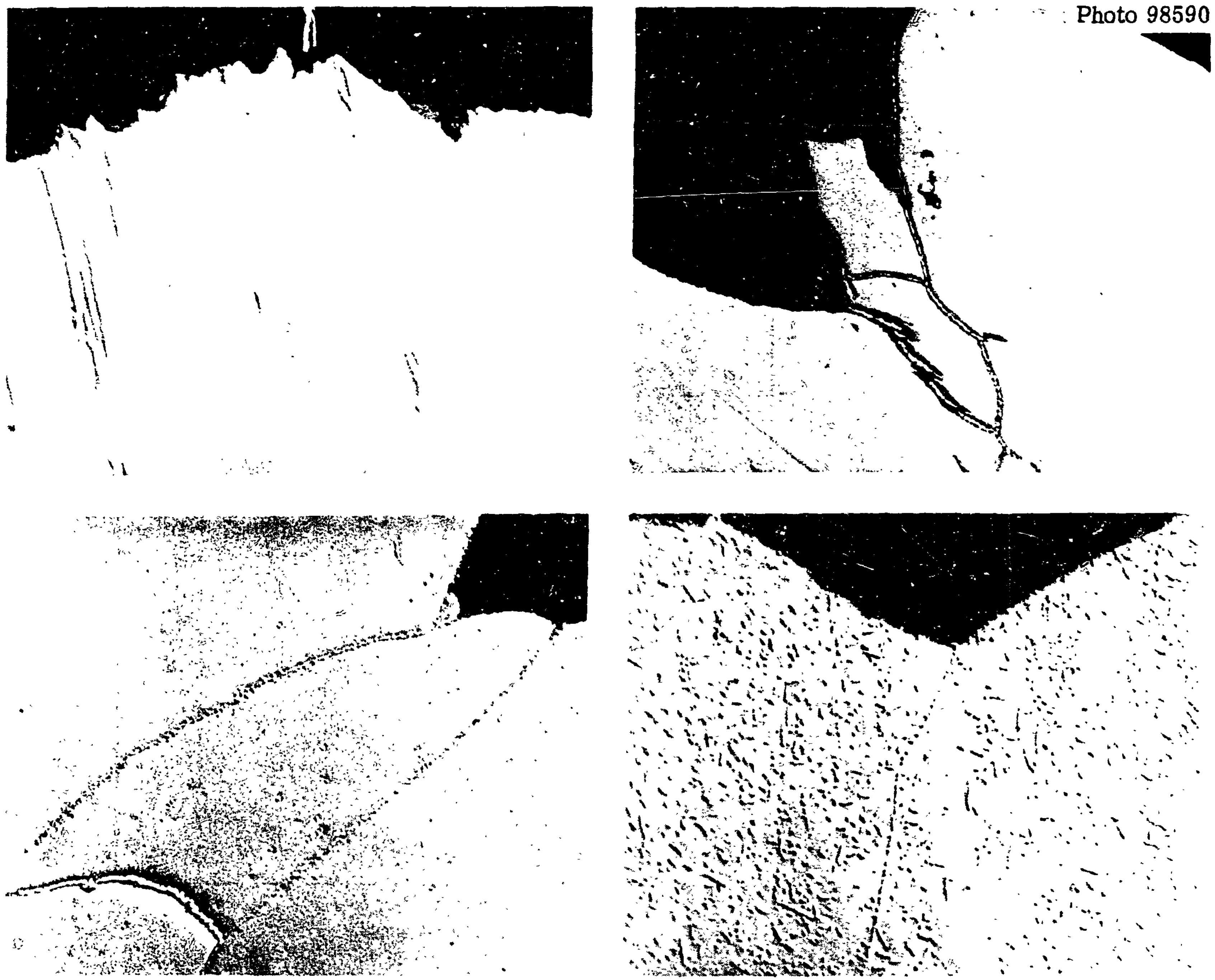

Fig. 14. Tensile Fractures of L-605 Aged at 760°C and Tested at 540°C. (a) Solution annealed at 1050°C for 1 hr; (b) aged at 50 hr; (c) aged at 500 hr; and (d) aged at 4000 hr. Potentiostatically etched. 1000×. Reduced 12%.

L-605 aged for 500 hr at 870, 815, and 760°C are compared in Fig. 15. It is quite evident that the morphology of the principal phase changes with temperature.

Specimens aged at 870°C for 4000 hr were heated to 1110°C for 200 sec to simulate reentry heating. Results of subsequent tensile tests at 1080°C showed no recovery of ductility. The solutioning rate is too sluggish to promote ductility recovery in 200 sec.

CONCLUSIONS

Prolonged exposure of L-605 in vacuum, argon, or air at temperatures ranging from 700 to 900°C will result in severe alloy embrittlement. The maximum rate of embrittlement occurs at 870 to 900°C and always within the initial 50 to 100 hr. At lower temperatures the embrittling rate is much less, but maximum loss of toughness occurs within the initial 500 hr. The principal embrittling reaction is the precipitation of the Laves phase, Co_2W. However, at lower aging temperatures of 650 to 800°C an added inducement to embrittlement may well be the precipitation of carbides ($M_{23}C_6$, M_6C, and M_7C_3), α-Co_3W, and β-Co_3W. At 760°C the greatest loss of toughness occurred during the initial 50 hr. The loss of toughness for longer aging times was only slightly less than for specimens aged at 870°C. Therefore, the ability of L-605 to sustain impact without failure subsequent to an aging exposure at 750 to 900°C for periods greater than 50 hr is severely reduced because fracture can occur at low impact energies.

The results of tensile tests at room temperature and 540°C showed that aged L-605 suffered a severe loss of tensile ductility. All fractures of aged material were totally intergranular because of grain boundary precipitation. At lower aging temperatures, sufficient tensile ductility was retained at 540°C for application at and above this temperature. However, severe loss of ductility was exhibited at room temperature. A brief reheating at 1110°C, as might be expected on reentry, did not restore ductility.

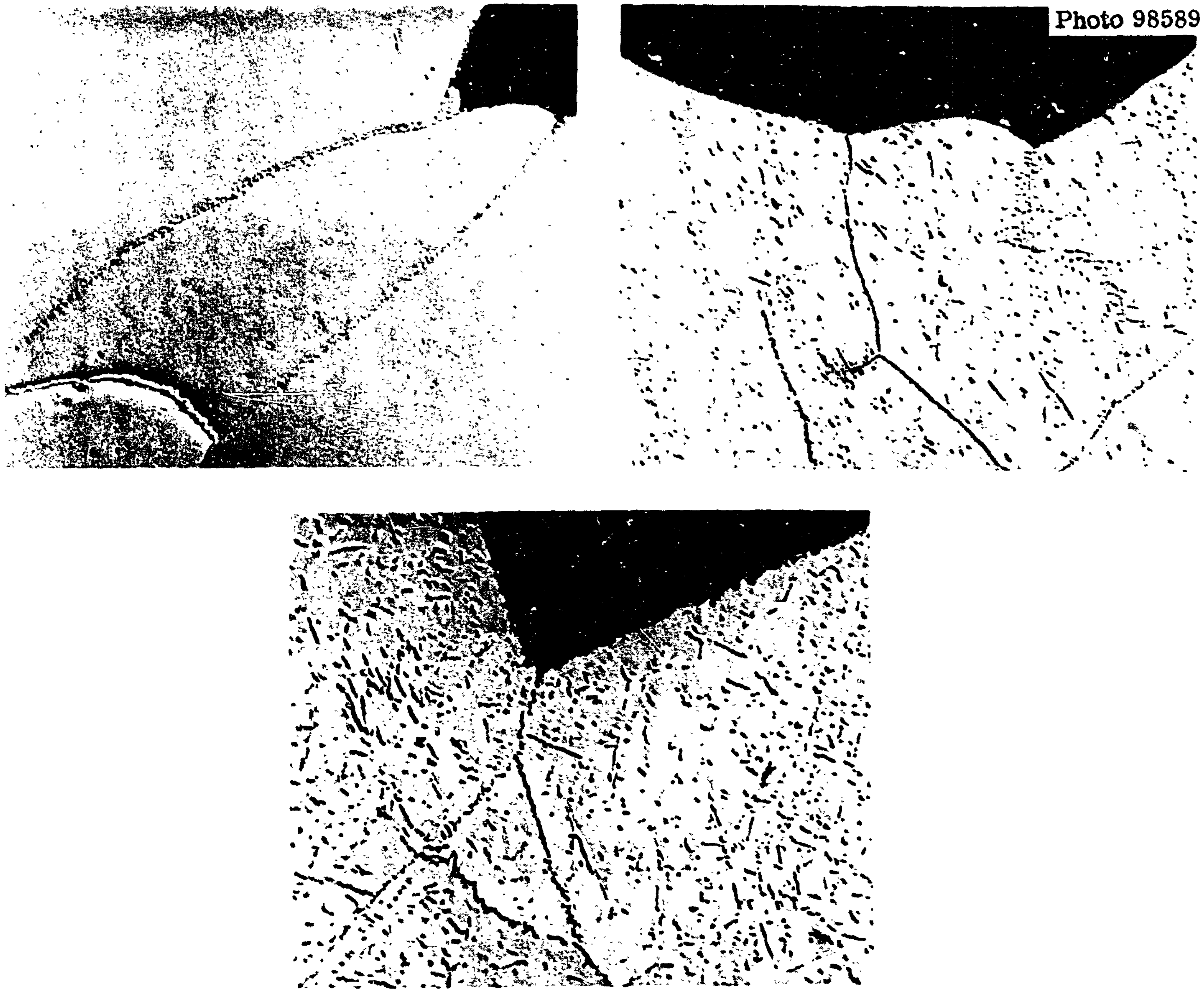

Fig. 15. Tensile Fractures and Microstructures of L-605 Aged for 500 hr and Tested at 540°C. (a) Aged at 760°C, (b) aged at 815°C, and (c) aged at 870°C. Potentiostatically Etched. 1000×. Reduced 5.5%.