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# Stress Corrosion Cracking in Quenched and in Underaged U-6 WT. % Nb

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# STRESS CORROSION CRACKING IN QUENCHED and in UNDERAGED U-6 WT.% Nb

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Chemical Metallurgy

## ABSTRACT

Two types of stress corrosion cracking occur in U-6 wt.% Nb. Intergranular (anodic dissolution) cracking occurs in aqueous $Cl^-$ environments. Transgranular (oxide stress rupture) cracking occurs in gaseous environments containing oxygen. Aging the quenched alloy at 200°C for 2 hours doubles the yield strength but only slightly lowers the stress corrosion cracking threshold.

## ACKNOWLEDGMENT

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INTRODUCTION

Uranium alloys are susceptible to stress corrosion cracking in [illegible] environments such as laboratory air and the U-[illegible] wt.% Nb alloy [illegible]. This investigation was undertaken to determine which species [illegible] is responsible for stress corrosion cracking and to define the [illegible] cracking threshold for U-[illegible] wt.% Nb in the as-quenched condition and in a low temperature aged condition, 200°C for 2 hours.

In the equilibrium condition the U-[illegible] wt.% Nb alloy [illegible] phase mixture of essentially pure $\alpha$ uranium and [illegible] with an approximate composition of $UNb_3$. [illegible] martensitically to a metastable $\alpha''_c$ [illegible]. [illegible] this $\alpha''_c$ structure in the [illegible] range [illegible] coherent precipitates which cause substantial strengthening [illegible] aging results in decomposition of the $\alpha''_c$ structure to [illegible] phases causing a loss of strength.

Figure 1 is a typical photomicrograph of the $\alpha''_c$ structure [illegible]. The grain boundaries outline the prior gamma grains. [illegible] $\alpha''_c$ [illegible] can be faintly seen within these prior gamma grains.

The corrosion resistance of uranium [illegible] increases as the [illegible] concentration increases. [illegible] of hydrogen generation [illegible] water [illegible] two orders of magnitude in [illegible] mixture [illegible]. The increase in internal hydrogen gas in the U-Nb alloy was negligible [illegible] from environment and was always [illegible] mixtures. Therefore, the rate of $H_2$ generation provides a reasonable estimate of the metal-$H_2O$ reaction rate. More recent data by Colloch[3] has shown that at room temperature the alloy is not very reactive. He obtained rates of $H_2$ generation of $< 6 \times 10^{-10}$ moles/cm$^2$ for 150-day tests in 10-90% R.H. air and 90-day tests in 10-90% R.H. $N_2$. Therefore, $H_2$ generation and corrosion are not major problems in ambient environments and the principle deterrent for the use of the U-6 wt.% Nb alloy is concern about its susceptibility to stress corrosion cracking.

## EXPERIMENTAL

The material used in this investigation was vacuum-arc melted and remelted. The alloy was upset forged and cross rolled at 850°C, homogenized at 1000°C for 12 hours and then water quenched from 800°C. The chemical compositions and mechanical properties of both the as-quenched and quenched and aged material are shown in Tables I and II, respectively.

The stress corrosion specimens were fatigue precracked single-edge-notch specimens (5.50 cm x 1.00 cm x 0.51 cm). The stress intensity, which is the mechanical driving force for stress corrosion crack propagation, for this type of specimen is defined as[4]

$$K_I = \frac{Pa^{\frac{1}{2}}}{BW} \left[1.99-0.41\,(a/W) + 18.70\,(a/W)^2 - 38.48\,(a/W)^3 + 53.85\,(a/W)^4\right],$$

where

$K_I$ = tensile mode stress intensity

$P$ = load

$a$ = crack length

$B$ = specimen thickness (0.51 cm)

$W$ = specimen width (1 cm)

with this specimen plane strain conditions were [illegible] stress intensity of the aged material in 50 ppm $Cl^-$ solution [illegible] thickness was less than the required

$$2.5\left(\frac{K}{\sigma_{y.s.}}\right)^2$$

for the other thresholds.

The 100% R.H. air environments were obtained by enclosing the specimen in a polyethylene bag containing water at the bottom, and the dry environment was obtained by substituting a $CaSO_4$ desiccant for the water. [illegible] environments were allowed to equilibrate [illegible] hours before the specimens were loaded. The 50 ppm $Cl^-$ environment was contained in a plastic [illegible] the specimen. The pure gas tests were conducted in [illegible] chambers. Mylar shims and filled epoxy coated metal pins were used to electrically isolate the specimen from the fixtures in all of the tests.

## RESULTS AND DISCUSSION

Tests were conducted on as-quenched U-6 wt.% Nb in several pure gases to determine which constituent of the air is responsible for stress corrosion cracking. The data, Table III, show that crack propagation will occur in $O_2$ and $H_2$ but not in $H_2O$ and $N_2$. $O_2$ has been identified as being responsible for the stress corrosion cracking of several uranium alloys in air.[illegible] While crack propagation occurred in $H_2$, massive hydriding did not take place on the crack surface as it did when U-7.5 wt.% Nb-2.5 wt.% Zr[illegible] and U-0.75 wt.% Ti were exposed to $H_2$.[7] There was evidence of hydride formation on the U-6 wt.% Nb specimens, but with the other alloys several millimeters of specimen along the crack faces formed hydride and spalled.

[illegible] on [illegible] material, in wet and dry air showed that while cracking [illegible] in pure $H_2$ [illegible] cracking is enhanced by the addition of $H_2$ [illegible] environments. The threshold for cracking [illegible], $K_{SCC}$, is [illegible] $MN/m^{3/2}$ ([illegible] Ksi $\sqrt{in}$) in dry air and 22 $MN/m^{3/2}$ (20 Ksi $\sqrt{in}$) in [illegible] wet air. Figure [illegible] shows the results of these tests and of tests conducted in aqueous solutions with 50 ppm $Cl^-$. The threshold in the dilute $Cl^-$ solution is even lower than in wet air, 17 $MN/m^{3/2}$ (15 Ksi $\sqrt{in}$). Koger[10] also found that $Cl^-$ enhances stress corrosion cracking in the U-6 wt.% Nb alloy.

Tests were also conducted on U-6 wt.% Nb which was aged at 200°C for [illegible] hours after quenching. The data, shown in Figure 3, show that the cracking thresholds were lowered slightly by the aging treatment. The thresholds are [illegible] $MN/m^{3/2}$ (1[illegible] Ksi $\sqrt{in}$) for the 100% R.H. air environment and 1[illegible] $MN/m^{3/2}$ (1[illegible] Ksi $\sqrt{in}$) for a 50 ppm $Cl^-$ environment. These data show that a low temperature age which more than doubles the yield strength of this alloy lowers the cracking thresholds only about 3 $MN/m^{3/2}$ (~20%). The data also show that the ratio of $K_{SCC}$ to the overload stress intensity is the same for both conditions of material. This indicates the primary effect of heat treatment is simply to lower the toughness of the material.

Higher strength levels of U-6 wt.% Nb have been tested by Miller[11] in 100% R.H. air and by Koger[10] in $Cl^-$ solutions. Miller[11] reports thresholds of 22 $MN/m^{3/2}$ (20 Ksi $\sqrt{in}$) for specimens heat treated at 250°C for 3 hours (yield strength ~ 69 $MN/m^2$, 100 ksi) and at 254°C for 6.4 hours (yield strength ~ 800 $MN/m^{3/2}$, 117 ksi). Aging at higher temperatures results in a higher yield strength, but also lead to increased susceptibility to stress corrosion cracking. A threshold of ~13 $MN/m^{3/2}$ (12 Ksi $\sqrt{in}$) was obtained for material with a nominal yield strength of 1100 MN/m$^2$ (160 ksi). [illegible] quenched, low temperature aged material [illegible] and overaged material (600°C for [illegible] hours) is significantly [illegible] to crack initiation and propagation in Cl$^-$ solution than material [illegible] for [illegible] hours, 500°C for 3 hours [illegible] hours.

The data on the effect of the heat treatment on the stress corrosion [illegible] of U-6 wt.% Nb show that the alloy can be aged to [illegible] improvement in strength with no significant loss in [illegible] resistance. However, at peak strength (intermediate aging temperatures) the alloy is very susceptible to stress corrosion cracking. [illegible] the overaged condition, which is resistant to stress corrosion cracking, would be expected to be more susceptible to general corrosion because of the formation of the α phase; however, this type of treatment [illegible] combination of high strength and stress corrosion cracking resistance [illegible] served in this alloy. Similarly overaging also increases the resistance to stress corrosion cracking in several other uranium alloy systems, including U-Ti,[12] U-Mo,[13] U-Nb[14] and U-Nb-Zr.[15]

The U-6 wt.% Nb fails by a different fracture mode in air [illegible] Cl$^-$ solutions. In Cl$^-$ solutions intergranular stress corrosion fracture is observed while in air environments (both wet and dry) fracture occurs by a quasi-cleavage mode. Figure 4 shows the intergranular mode while Figure [illegible] shows the transgranular stress corrosion cracking mode. The stress corrosion fracture modes can be discerned from the fatigue and overload regions both macroscopically and microscopically. Macroscopically, the stress corrosion region is darkened and can be easily identified. Fatigue and overload [illegible] are shown in Figures [illegible] and [illegible], respectively, and can be seen [illegible] completely from stress corrosion fractures. The fractographs shown in Figures [illegible] are characteristic of both the as-quenched and quenched and aged material.

The U-6 wt.% Nb alloy's stress corrosion cracking behavior is similar to that of other dilute "rich" uranium alloys. The transgranular and intergranular cracking are attributed to different cracking mechanisms in this class of uranium alloys. Intergranular cracking is attributed to an anodic dissolution process[6] and requires $O_2$, $H_2O$ and $Cl^-$ to initiate.[7] In the absence of $Cl^-$ a slower transgranular stress corrosion cracking process occurs. This type of cracking is attributed to an oxide stress rupture mechanism wherein the applied stress is supplemented by the stresses from an anisotropic oxide.

## CONCLUSIONS

1. Two types of stress corrosion cracking occur in U-6 wt.% Nb. Intergranular (anodic dissolution) cracking occurs in $Cl^-$ solutions while transgranular (oxide stress rupture) cracking occurs in $Cl^-$ free environments containing $O_2$.
2. While U-6 wt.% Nb will not crack in pure $H_2O$ the addition of $H_2O$ to environments enhances stress corrosion crack propagation.
3. Aging material at 200°C for 2 hours doubles the yield strength but only slightly increases the susceptibility to stress corrosion cracking.

TABLE I

Chemical Composition Including Major Impurities

| | Nb (wt%) | Fe | Si | C | $N_2$ | $O_2$ | $H_2$ |
|---|---|---|---|---|---|---|---|
| | | ppm | | | | | |
| As-quenched Material | 6.12 | 43 | 27 | 59 | 24 | 37 | 1.[illegible] |
| Aged Material (200°C/2 hours) | 6.23 | 18 | 20 | 64 | 2[illegible] | 31 | 1.[illegible] |

TABLE II

Mechanical Properties*

| | Yield Strength ($MN/m^2$) | Yield Strength (Ksi) | Ultimate Tensile Strength ($MN/m^2$) | Ultimate Tensile Strength (Ksi) | Elongation in 4 diameters (%) |
|---|---|---|---|---|---|
| As-quenched Material | 170 | 24 | 7[illegible]0 | 1[illegible] | 31 |
| Aged Mate (200°C/2 | 360 | 5[illegible] | 500 | 1[illegible] | [illegible] |

*Averages of specimens from several locations and orientations in the plate, after Kirby.[4]

TABLE III

The Time-to-Failure of [illegible] As-quenched U-[illegible] wt.% [illegible]

Loaded at ~37 MN/m$^{3/2}$ (34 ksi $\sqrt{\text{in}}$) in Pure Gases

| Environment | Pressure (torr) | Time-to-Failure* (hours) |
|---|---|---|
| $O_2$ | [illegible] | [illegible] |
| $H_2$ | [illegible] | [illegible] |
| $H_2O$ | [illegible] (100% R.H.) | No crack growth in [illegible] |
| $N_2$ | [illegible] | No crack growth in [illegible] |

*Averages of duplicate tests.

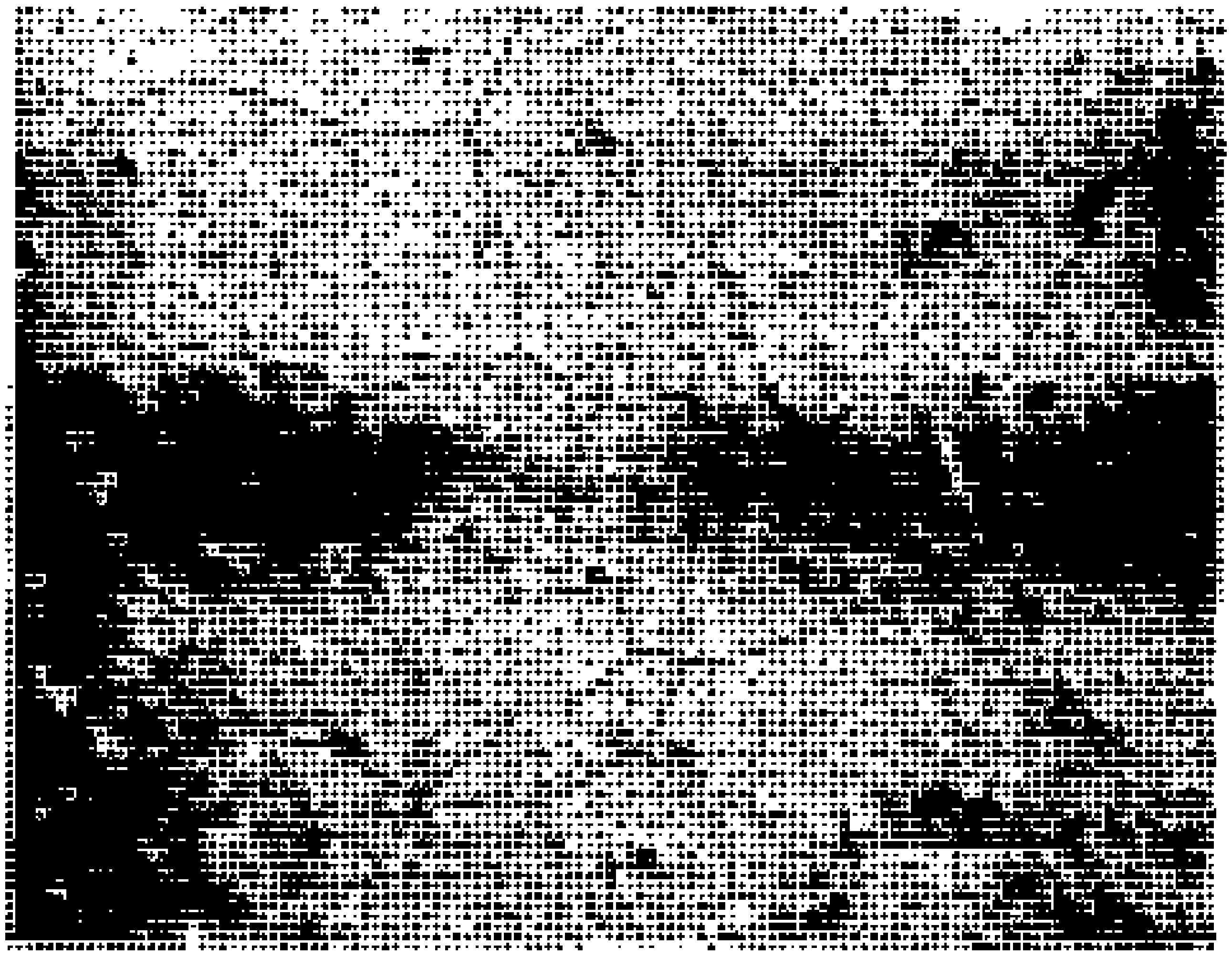

Figure 1. Microstructure of as-quenched U-6 wt.% Nb.

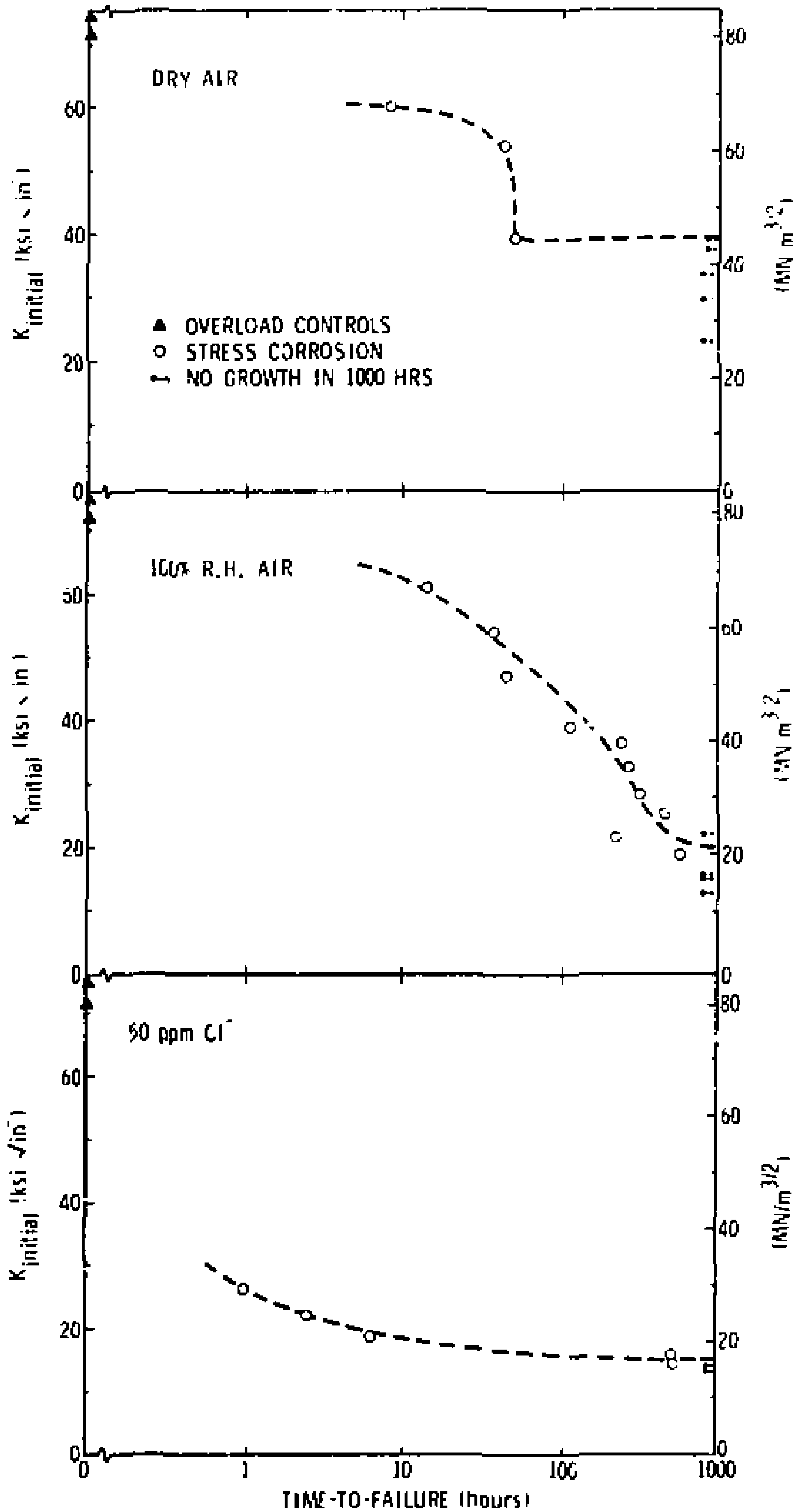


*Figure 2.* The relationship between the initial stress intensity and the time-to-failure for as-quenched U-6 wt.% Nb.

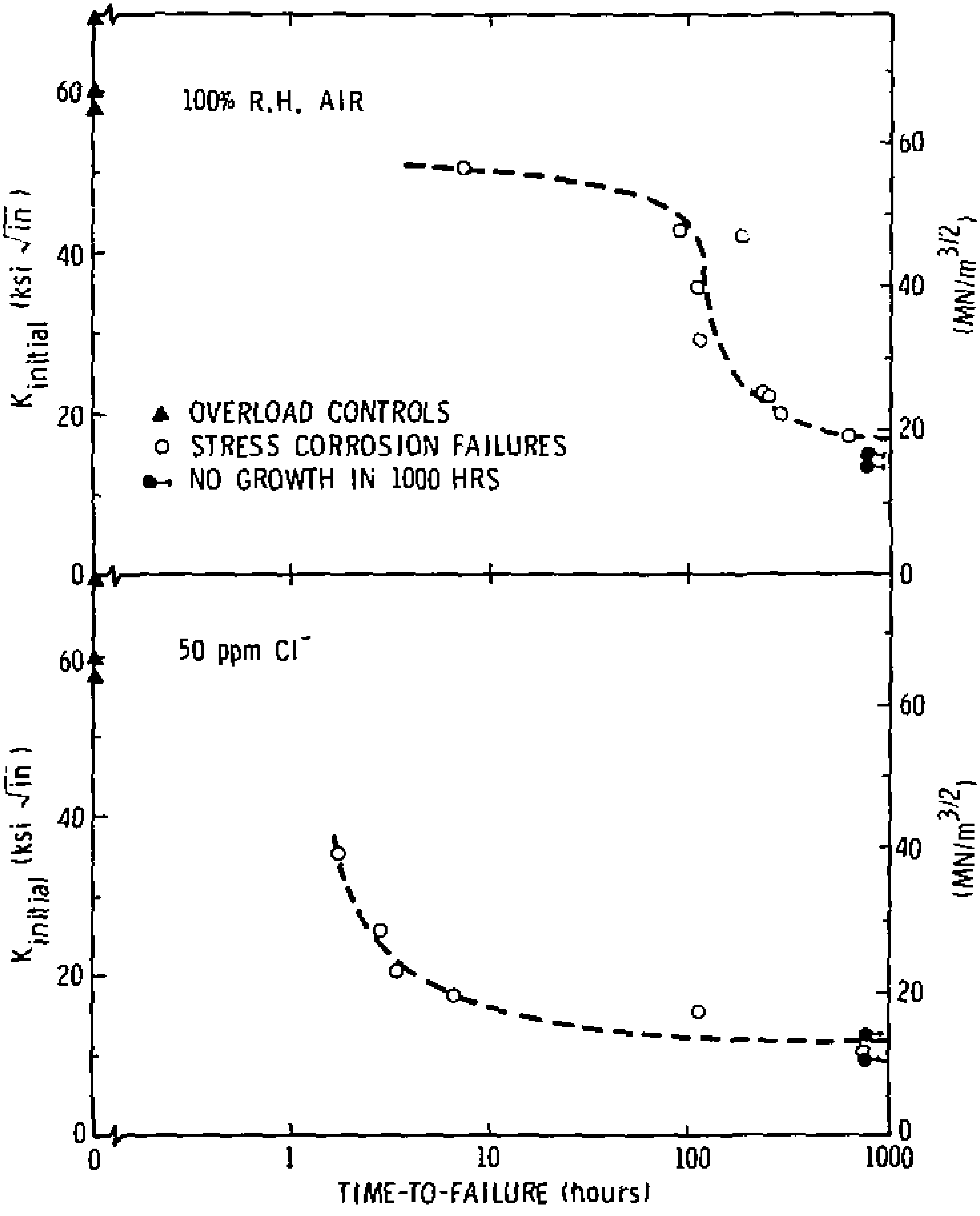


Figure 3. The relationship between the initial stress intensity and the time-to-failure for quenched and aged (200°C/2 hours) U-6 wt.% Nb.

Figure 4. An intergranular stress corrosion fracture surface of U-6 wt.% Nb - characteristic of as-quenched and aged material tested in dilute $Cl^-$ solutions.

Figure 5. A quasi-cleavage stress corrosion fracture surface of U-6 wt.% Nb - characteristic of as-quenched and aged material tested in dry air and in 100% R.H. air.

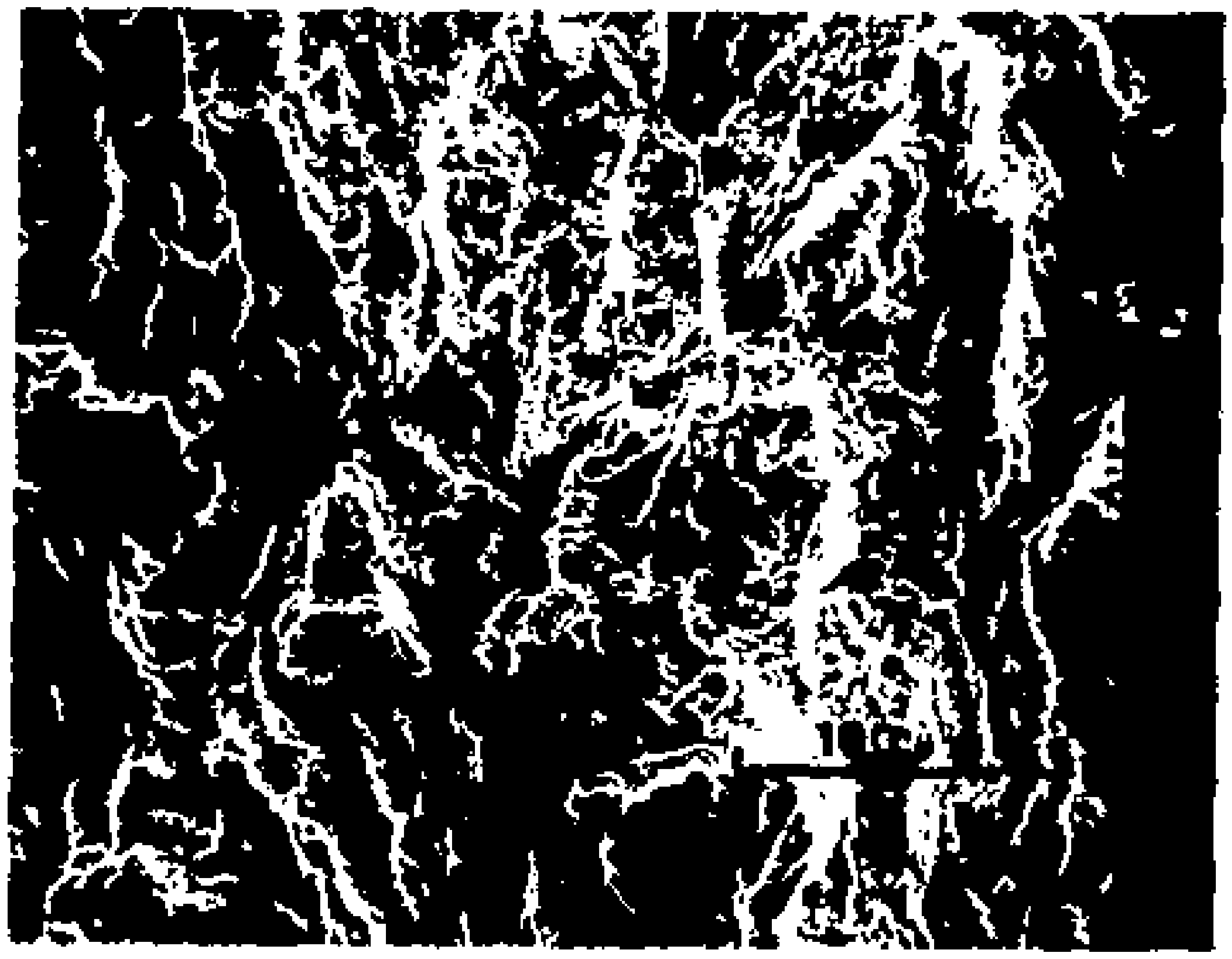

Figure 6. A fatigue fracture surface of U-6 wt.% Nb - characteristic of both as-quenched and aged material.

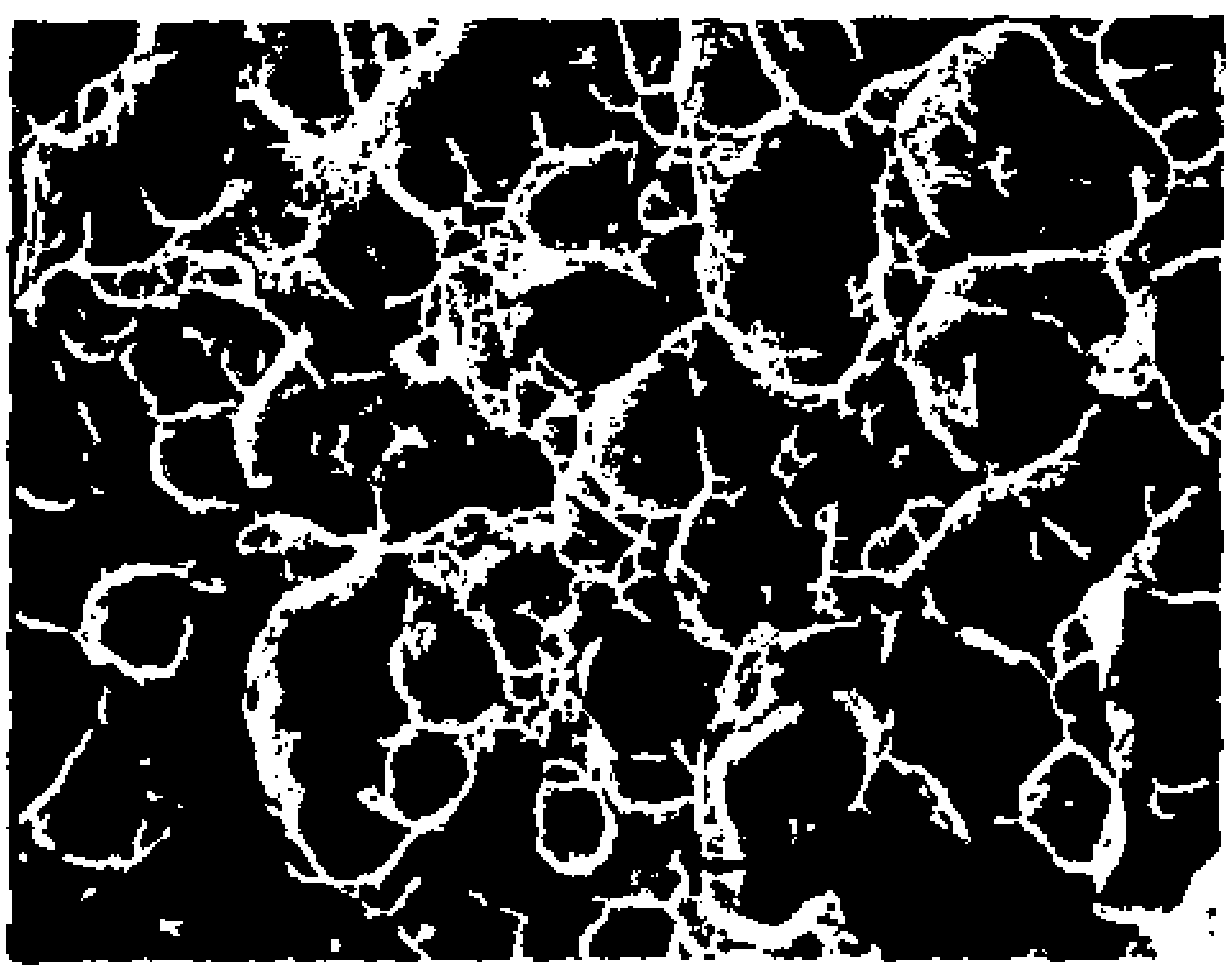

Figure 7. An overload fracture surface of U-6 wt.% Nb - characteristic of as-quenched and aged material.

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