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URANIUM-10 w/o MOLYBDENUM ALLOY

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STRESS-CRACKING IN THE URANIUM-10 w/o MOLYBDENUM ALLOY

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ABSTRACT

It was found that the U-10 w/o Mo alloy is subject to delayed failure in air. Oxygen was determined to be the embrittling agent. The critical stress, or threshold stress, for delayed failure is as low as 37,000 psi. This threshold stress increases with decreasing carbon content. The 0.2% offset yield strength of this alloy, in the dynamic tensile test, is typically 125,000 psi.

INTRODUCTION

Production of sound welds in the uranium-10 w/o molybdenum alloy is difficult. Both transverse and longitudinal cracking occur in the weld zone. In general, fracture takes place shortly after welding, but in some cases the welds failed after several days exposure to laboratory air. In subsequent studies it was found that relief of residual stresses by postheating the welded plates prevented cracking. However, postheating is not possible in many applications. Attempts to accomplish stress relief by preheating the weld plates were not successful.

The uranium-10 w/o molybdenum alloy can be either hot or cold worked, but cold working is much more desirable because the alloy is not oxidation resistant even at moderate temperature. Fracture also occurs in cold-worked parts unless the material is annealed soon after fabrication. Inspection of cold-rolled plates after storage in air for a few days revealed extensive cracking. In conjunction with the above observations, Y-12 reported failures of uranium-8 w/o molybdenum parts stressed in air,¹ and workers at AERE Harwell found severe surface cracking on specimens tested in tension.²

The apparent susceptibility to cracking casts serious doubt as to the usefulness of the uranium-10 w/o molybdenum alloy. Therefore, a program

was undertaken to determine the cause of the phenomenon, and to examine possible ways to eliminate or at least reduce the problem. This report presents results of the investigation to date.

SUMMARY AND CONCLUSIONS

The uranium-10 w/o molybdenum alloy is highly susceptible to cracking, and hence ultimate failure, when stressed in air. Tests conducted in various environments show that oxygen is responsible for the cracking. In this alloy the elongation in a stress-strain test is sensitive to strain rate, and the maximum ductility is obtained at a rate of about 0.05 in./in./min. Tests at both faster and slower strain rates result in lower elongation. Delayed failure was observed at a stress as low as 37,500 psi (30% of the yield stress). The critical stress appears to be dependent on carbon content, but not on grain size. Attempts to protect the alloy by electrolytic or vapor-deposited coatings were not successful. Small additions of niobium to the binary alloy do not alleviate the cracking problem. The critical stress for a uranium-8 w/o molybdenum-1 w/o titanium alloy was determined to be 108,000 psi. This value is the highest for all the materials tested that demonstrated the stress-cracking.

DISCUSSION

Two types of tests were employed to evaluate the properties of the uranium-10 w/o molybdenum alloy. A static tensile test was used to determine the load-bearing capability of the alloy below the yield stress, and the normal dynamic tensile test was used to ascertain the yield stress, ultimate tensile stress, and elongation, as well as strain-rate effects and the work-hardening characteristics of the material.

A variety of sets of specimens, each set with a different thermal and mechanical history, were tested in this study. However, the final treatment for all specimens was a gamma anneal followed by quenching. The impurity content was about the same for the different types of specimens except for carbon content. The effect of carbon will be discussed separately.

A series of tensile tests was conducted on specimens at different strain rates in both air and vacuum. These samples had a carbon content of about 400 ppm. A typical stress-strain curve is shown in Fig. 1. As can be seen from the curve, the ultimate tensile strength was reached shortly after

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Fig. 1. Engineering stress-strain curve for U-10 w/o Mo alloy.

yielding; thereafter the load decreased until fracture took place. The deformation was highly localized, and it occurred almost exclusively in the narrow region which necks down during testing. It seems that once yielding is initiated it is much easier to sustain plastic flow in that zone than to initiate yielding elsewhere in the specimen. This alloy does not have good work-hardening characteristics. The elongation of specimens tested in air at various strain rates are shown in Fig. 2. The ductility increased with decreasing strain rate to a deformation rate of about 0.05 in./in./min, and then the elongation decreased to almost zero at very low strain rates. The ductility of this alloy in air is strongly dependent on strain rate. Specimens tested at rates exceeding 0.05 in./in./min did not show any surface cracking or an oxidized rim of material at the surface as reported by Harwell for a strain rate of 0.06 in./in./min. However, at rates below 0.05 in./in./min, surface cracking was observed, and it became progressively more severe as the strain rate was decreased. Microstructural examination of specimens exhibiting surface cracking revealed the cracks to be primarily transgranular (Fig. 3). The edge of the sample is parallel to the stressing direction; thus, the cracks are perpendicular to the tensile axis. The branching and forking type of crack is similar to those observed in stress corrosion cracking of some high-strength steels. It should be pointed out that many specimens for this study were rejected because of extensive cracking at identification numbers stamped on the specimens by the supplier.

In order to complement the tests performed in air, a series of tests were run in vacuum. These data are also shown in Fig. 2. Elongations increased to the slowest strain rates that were used. However, the characteristics of the stress-strain curve were unaltered, indicating that the absence of work hardening is a basic property of this alloy. No surface cracking was found on any of the vacuum-tested samples.

It is evident from the above test results that some constituent in the air was responsible for the surface cracking and loss of ductility in the slow strain-rate tests. It is also apparent that at greater times under load (i. e., low strain rates) the effect is more pronounced. The next step was to determine what gas in the air caused the cracking. Another series of tensile tests was conducted on specimens in the pure gases which are the major constituents of air. After the test chamber was evacuated to about 10^{-5} mm

Fig. 2. The effect of strain rate on ductility.

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Fig. 3. Surface cracking observed on low strain-rate test specimens (100 $\times$).

Hg, it was back-filled with a specific gas, and then the tensile test was started. All tests were conducted at a strain rate of 0.0005 in./in./min. The results of these tests are reported in Table I. It is immediately obvious that oxygen is the gas responsible for the embrittling effect observed in uranium-10 w/o molybdenum alloy. It is also of interest to note that the embrittlement is more severe in dry air than in laboratory air with a controlled 50% relative humidity. Adsorption of water vapor on the crack surface may inhibit oxygen from reaching the crack front, thus retarding the extension of the crack. Further work on the effect of humidity on the ductility of the alloy is needed to clarify this point.

Table I. Tensile properties in various environments.

Environment	Elongation ^a (%)	Yield stress (psi)	Ultimate tensile stress (psi)
Vacuum	19.0	105,000	108,000
Air	5.0	97,600	103,000
Dry air	1.3	90,100	93,400
Dry N ₂	19.3	105,300	109,000
Dry O ₂	1.2	92,200	94,000
Dry CO ₂	20.0	98,400	103,500

^aAll tests conducted at $\dot{\epsilon} = 0.0005$ in./in./min.

The cracking observed in the welded plates and cold-rolled strip indicated that residual stress in conjunction with oxygen in the air can cause failure. This phenomenon is called delayed failure, and it is also typical of hydrogen and liquid-metal embrittlement. In most cases a critical minimum stress exists below which failure will not occur. In order to determine the critical stress for the lot of specimens used in the stress-strain work, a series of static tensile tests was made. In these tests a static tensile stress, which was less than the observed yield stress, was applied to the specimen, and the time to failure was recorded. The stress was progressively lowered until the critical stress was established. The results of these tests are shown in Fig. 4. The critical stress for an alloy with 400 ppm carbon is around 30% of the yield strength. This corresponds to a stress of 37,500 psi. The surface of all specimens was covered with macroscopic cracks, but

Fig. 4. Delayed failure characteristics of material containing 400 ppm carbon.

more extensive cracking occurred in the specimens tested at the lower stresses. The fact that each sample contained numerous cracks other than the one which caused failure indicated that crack growth, not crack nucleation, was the controlling process. This also could be deduced from the branching type cracks found in the tensile test samples. This means that a crack propagates into ductile metal and then stops, and will only propagate again after the material at the crack tip is embrittled by further oxygen supply from the air. The fracture surface on static tensile-test specimens consists of two distinct regions. In one area fracture has occurred by a cleavage mechanism and in the other area by shear. The fracture process can probably be described as follows: Take, for example, a specimen loaded to 60% of the yield stress. Once a crack nucleates, it propagates a short distance into the specimen by cleavage, but is arrested in "good" material. Oxygen is then supplied to the crack front and after a period of time the surrounding area becomes embrittled and the crack propagates once more, but it is again stopped. This process continues until approximately 40% of the load-bearing area has cracked. Now the applied stress is about equal to the ultimate tensile stress of the uncracked area and the final fracture takes place in a ductile manner.

Three other groups of samples of the uranium-10 w/o molybdenum alloy were available for testing. The primary differences between the various types of sample were grain size and carbon content. Because only a limited number of specimens of these groups were available and because parts in service are rarely stressed beyond the yield point, the stress-cracking behavior of these samples was evaluated by static tensile tests (Fig. 5). The metallic impurity content and nitrogen, oxygen, and hydrogen content were essentially the same for all groups of specimens. Consequently, the differences in grain size and carbon content permitted evaluation of the effect of these variables on the stress-cracking phenomena exhibited by this alloy.

It has been demonstrated that in many cases the fracture strength of a material is related to grain size by the Stroh-Petch relationship $\sigma_F = \sigma_0 + Kd^{-1/2}$ where σ_F is the fracture stress, σ_0 and K are constants, and d is the mean grain diameter. Thus, small grain size should promote better fracture strength. Examination of the fracture stress at constant time for the various types of specimens does not show any consistent trend

Fig. 5. Delayed failure curves for various grain size and carbon contents.

and it may be tentatively concluded that grain size, at least for the values available in these experiments, has no appreciable effect on the fracture process.

Work at Watertown Arsenal Laboratories has shown that carbon content can significantly reduce the ductility of uranium-8% molybdenum alloys, and it appears from Fig. 5 that carbon content is also an important variable in the stress-cracking problem. The trend, as indicated by the data, is to raise the critical stress for delayed failure by reducing the carbon content of the alloy. If carbon content is the exclusive parameter involved, it is interesting to speculate whether the stress-cracking phenomenon could be eliminated by using carbon-free material. Further testing of ultra-high-purity material is needed.

Evidence that oxygen in the surrounding atmosphere is responsible for the failure of uranium-10 w/o molybdenum parts stressed at room temperature leads to the conclusion that the oxide of this alloy provides little if any barrier to oxygen penetration. Hence, two methods were employed in an attempt to eliminate the cracking problem: (1) coating, and (2) alloying. In the first case the protective coating should prevent access of oxygen to the surface of the alloy, while in the second case it was hoped that the oxide of the ternary alloy might provide a more sufficient barrier to oxygen penetration.

Tensile bars of the material containing 400 ppm carbon were coated by electrodeposition of nickel, or by vapor deposition of copper or copper and gold. The nickel layer was 3 mils thick and tightly adherent, but the specimens stressed at 80% of the yield stress failed in about 45 minutes, or approximately one-half the time of the uncoated specimens. On the supposition that no oxygen reached the substrate, it was assumed that hydrogen pick-up during electroplating was responsible for failure. The hydrogen content before and after plating is being determined to verify this assumption. The result that hydrogen has a deleterious effect on this alloy is not too surprising since it is well documented that the mechanical properties of b. c. c. metals are sensitive to interstitials and that some metals, particularly iron, are extremely sensitive to hydrogen content. In addition, previous data (UCRL-7771) have shown that the reduction of hydrogen content in the uranium-10 w/o molybdenum alloy by vacuum annealing can double the impact strength. Thus, it would seem that any electrolytic coating method must be ruled out as a way of protecting the alloy.

Tensile bar specimens to be vapor coated were first cathodically cleaned and then, without exposure to oxygen, a thin coating was evaporated onto the cold substrate. Specimens statically stressed to 80% of the yield stress failed in approximately 30 minutes. Again no improvement was obtained. Microscopic examination of this coating revealed a tightly adherent coat of 1 to 3 mils in thickness, but numerous cracks penetrated the coating from its surface to the uranium-10 w/o molybdenum substrate. Consequently the pre-existing cracks not only created points of stress concentration but a path for oxygen to reach the alloy. It is apparent that condensation and subsequent cooling of the copper or copper and gold creates large stresses from thermal contraction, and that these stresses are of sufficient magnitude to crack the coating. Improvement of the coating procedure (e. g., heating of the substrate) might provide crack-free coats.

Only four bars of a uranium-8 w/o molybdenum-2 w/o niobium alloy were available. Because of the lack of data on this system, and the limited number of specimens, it was decided to conduct stress-strain tests in air with these specimens. In this way the tensile properties for this alloy could be obtained, and by varying the strain rate, the susceptibility of this alloy to stress cracking could be determined by comparison of the elongations obtained at a fast and slow rate. If the cracking behavior is absent, the elongation at the slow rate should be equal to or greater than the elongation at the fast rate; this was the case for the uranium-10 w/o molybdenum material tested in vacuum.

Conversely, the elongation at the slow rate should be much less than that at the fast rate, as in the air tests of the uranium-10 w/o molybdenum alloy (Fig. 2), if the cracking problem is still present in the ternary alloy. At a strain rate of 0.05 in./in./min, the yield stress, ultimate tensile stress, and elongation are, respectively, 122,000 psi, 132,000 psi and 16%. These values are almost the same as those for the binary uranium-molybdenum alloy. The stress-strain curve for this alloy is likewise similar to the uranium-10 w/o molybdenum alloy with little exhibition of any work hardening. The yield stress, ultimate tensile stress, and elongation at a strain rate of 0.0005 in./in./min were 91,000 psi, 95,000 psi and 2.0% respectively. The elongation of 2.0% at the low strain rate can be compared with 5.0% obtained for the binary alloy in air at an equivalent strain rate. On this basis it would seem that small additions of niobium offer little hope for elimination

of the cracking problem. However, additional static tensile testing is required to establish the critical stress for the alloy.

The critical stress for a uranium-8 w/o molybdenum-1 w/o titanium alloy was determined by static tensile tests. The value for this alloy was found to be 108,000 psi. This can be compared with the optimum value of 87,500 psi for the binary uranium-10 w/o molybdenum alloy containing 65 ppm carbon. The carbon content of the alloy containing titanium was 15 ppm. Previously it was demonstrated that carbon content appears to be an important variable in the stress-cracking process. Thus, it is not certain whether the higher critical stress in the uranium-8 w/o molybdenum-1 w/o titanium is due to the lower carbon content or increased resistance to oxygen penetration by the presence of the titanium. Nevertheless, the critical stress of 108,000 psi is the highest observed in this study.

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