

§19. High Temperature Creep of Ultra-Fine Grained, Particle Dispersed V-V-W-TiC Alloys

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The issues of vanadium (V) and its alloys for fusion reactor structural applications are decrease in strength at high temperatures and radiation embrittlement due to neutron and He irradiations. In order to overcome the issues, V alloys with very fine grains and nano-dispersoids were developed by powder metallurgical (P/M) methods utilizing mechanical alloying (MA) and hot isostatic pressing (HIP). The fabricated alloys exhibited good resistance against neutron irradiation, however their high temperature strengths significantly decreased above 1073~1123K due to grain boundary sliding caused by a very fine grain size. Recent results on constant rate tensile tests for V-1.6Y-8W-(0.4, 0.8)TiC (wt%) alloys showed that the alloys exhibit higher strength up to around 1273K than solution hardened V-4Cr-4Ti alloys. High temperature structural applications of materials require their evaluation of response not only to short-time tensile tests, but also to long-time creep tests. Since long-time creep properties may be affected more significantly by grain boundary sliding than short-time tensile properties, it is very important to study the effects of grain size on the long-time creep behavior of the V-1.6Y-8W-(0.4, 0.8)TiC alloys.

In this study V-1.6Y-8W-0.8TiC specimens with three different grain sizes were prepared. In order to examine the long-time creep properties of the three kinds of V-1.6Y-8W-0.8TiC specimens, we designed and prepared a testing fixture applicable to the creep testing machine at NIFS. In addition, we measured the strain rate dependence of yield stress of the three kinds of specimens at 1073K, the highest testing temperature available for the present creep machine, to determine the appropriate creep testing conditions. By using the fixture and creep machine combined with the measured and already obtained data, we conducted a preliminary creep test to confirm the reliability of the creep test results.

An alloy with the composition of V-1.6Y-8.5W-0.8TiC and relative density of approximately 99.7% was fabricated from commercially available powders of pure V, Y, W and TiC by the P/M method utilizing MA and HIP. For the MA process three mutually perpendicular directions agitation ball milling with vessels and balls made of TZM (Mo-0.5%Ti-0.1%Zr) was conducted for 50 h in a purified

H₂ atmosphere. The MA processed powders were HIPed at 1273 K and 200 MPa for 3 h, followed by annealing at 1473, 1573 and 1473 K for 1h in a vacuum. The annealed specimens were subjected to XRD, TEM observations, constant rate tensile tests and creep tests. The constant rate tensile tests were carried out at 1073 K at initial strain rates from 1.0×10^{-4} to $1.0 \times 10^{-2} \text{ s}^{-1}$ in a vacuum better than $3 \times 10^{-4} \text{ Pa}$. The creep tests were performed at 1073 K and 250 MPa. It should be noted here that the specimens used in this study are in the as-HIPed and annealed states without any plastic working. The main results obtained are as follows.

1. XRD patterns of the annealed specimens show distinct peaks of V, Y₂O₃, TiC, Y₂C, but no peaks of W. This indicates that the strength of the annealed specimens is mainly attributable to both precipitation hardening by Y₂O₃, TiC and Y₂C and solution hardening by W.

2. The average grain sizes are in the range of 580 ~ 1450 nm, which increases with increasing annealing temperature. The average diameters of the dispersoids, such as Y₂O₃ and TiC, are less than 100 nm.

3. The yield stresses, σ_y , of the annealed specimens depend on the initial strain rate, and the strain rate dependence decreases with increasing annealing temperature; 350~500 MPa for 1473 K anneal, 345~417 MPa for 1573 K anneal and 344~411 MPa for 1673 K anneal. The results suggest that the 1673K annealed specimens may exhibit the highest resistance to creep deformation which occurs at a very low strain rate.

4. The creep testing fixture for miniaturized specimens with the gauge section of 1.2 mm in width, 5.0 mm in length and 0.40~0.45 mm in thickness was newly prepared. The specimen thickness is 1.6~1.8 times as large as that used so far at NIFS and the new fixture enables specimen setting without grip-tightening so that the uniaxiality and alignment of the specimen are readily ensured.

5. On the basis of the above tensile-test results and the creep data on various V alloys obtained so far at NIFS by the same creep machine, the applied stresses at 1073 K in the creep tests were determined to be 100, 150, 200 and 250 MPa. A specimen annealed at 1473 K, which yielded the smallest grain size and thus may exhibit the lowest creep resistance, was subjected to preliminary creep tests at 1073 K and 250 MPa (= 115.9 N) in a vacuum of $\sim 1 \times 10^{-5} \text{ Pa}$. 20 minutes after applying the stress the specimen exhibited creep deformation with the steady state where the strain rate becomes a constant, $1.3 \times 10^{-5} \text{ s}^{-1}$, and elongation of 60%, followed by ductile fracture after 235 minutes. The creep curve obtained by the preliminary test was checked and judged to be reliable and therefore it was concluded that it is possible to evaluate the effects of grain size on the creep properties of ultra-fine grained and fine grained, precipitation and solution hardened V-1.6Y-8W-0.8TiC alloy by the present combination of the creep testing fixture and apparatus.