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Effect of precipitation and solution behavior of impurities on mechanical properties of low activation vanadium alloy

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Abstract

Since vanadium alloys work harden during rolling to produce plate and such processing results in anisotropic mechanical properties, a post rolling heat treatment is required to recover the material properties. The effect of heat treatment temperature on mechanical properties was investigated and it was confirmed that heat treatment at 1073 K would recover the work hardening and provide better mechanical properties. In addition, the precipitation behavior of Ti–C–O was studied using solution heat treated plate to observe changes in the mechanical properties. Fine precipitates formed in high density at 973 K, increasing the yield stress. At 1173 K, larger precipitates formed at low density which decreased the yield stress. The 1373 K heat treatment dissolved all precipitates and resulted in a higher yield stress than that at 1173 K.

1. Introduction

V–4Cr–4Ti has been designed and fabricated at the National Institute for Fusion Science as a low activation structural material for fusion devices [1,2]. It has been reported that impurities such as carbon, nitrogen and oxygen affect the mechanical properties strongly [3–5]. Since the mechanical properties are factors in the development of structural materials, some investigations have been carried out using base plates and weld joints [6,7]. Precipitation and solution behavior of the impurities depend on the heat treatment conditions, so, it is desirable to study the effect of impurities on the Vickers hardness and mechanical properties.

In this report, the results of tensile, hardness and Charpy tests of annealed plates after cold working and reheated materials after solution heat treatment will be presented. The effect of annealing temperature on the mechanical properties is explained and the desirable temperature for annealing is discussed. Also, precipitation and solution behavior of the solution heat treated plates are described.

2. Test material and experimental procedure

The material used was a V–4Cr–4Ti plate of NIFS-HEAT-2 which was rolled to 4 mm thick with the reduction ratio of 95% in cold work. The chemical composition in wt% is 3.46Cr–3.75Ti and it contains 62 ppm carbon, 116 ppm nitrogen and 139 ppm oxygen. The as-rolled plate was annealed in vacuum in the range of 873–1373 K for 1 h. To investigate the effect of heat temperature on the precipitation and solution behavior, another series of tests were performed. The as-rolled material was solution heat treated at 1373 K to recrystallize the cold worked microstructure, then reheating was conducted to precipitate or dissolve the impurities. The solution heat treatment was carried out for 1 h in

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vacuum and the reheating was performed in the temperature range of 873–1373 K for 1 h in vacuum. Specimen was put in Ta box and wrapped by Zr foil, then, heat-treated to reduce oxidization. The vacuum was kept at less than 10^{-4} Pa during the heat treatment.

To determine the effect of heat treatment temperature, the tensile and Vicker's hardness, Charpy testing along with SEM and TEM observations were carried out. The tensile specimen was a round bar of 2.6 mm in diameter and had a parallel part of 10 mm long. The tensile tests were carried out at room temperature. Two extensometers with an 8 mm gage length were attached to the specimen symmetrically and the average displacement was calculated to cancel the bending strain component so that the Young's modulus and yield stress were measured accurately. The stroke rate was controlled to be an initial strain rate of 5×10^{-4}/s. In the Charpy test, 1/3CVN specimens (3.3 mm $\times$ 3.3 mm in cross section) were used. The supporting span was 15 mm and the drop velocity was 4.5 m/s.

3. Results and discussion

The as-rolled plate showed remarkable anisotropic mechanical properties. For example, the tensile test results of the as-rolled specimens are shown in Fig. 1. L-direction designates the rolling direction and T-direction means the direction transverse to the L-direction. The tensile properties of material tested in the T-direction are brittle-like and the fracture strain at final fracture is very small. In addition, specimens tested in both directions did not show strain hardening, for the plate was heavily cold worked. The T-direction specimen showed little necking and the L-direction specimen displayed an elliptical fracture surface elongated in the transverse direction. Fig. 2 shows the results of tensile tests of the L-direction specimens heat treated at different temperatures. The specimen annealed at 873 K showed some strain hardening with a larger total elongation. However, there is not much change in tensile properties for annealing at temperatures over 1073 K and the fracture strain is consistently about 40%. The anisotropic deformation behavior was improved by 1073 K heat treatment. Fig. 3 summarizes the yield stress, the tensile strength, the fracture strain and the Vicker's hardness against heat treatment temperature. As the annealing temperature goes up, the hardness decreases and the yield stress and the tensile strength gradually drop down. On the other hand, the fracture strain increased rapidly between 973 and 1073 K. Therefore, these results indicate that recrystallization begins about 1073 K.

The Charpy test results are shown in Fig. 4. The specimens were oriented so that crack propagation occurred transverse to the rolling direction. Specimens heat treated at 1073 and 1173 K show a gradual decrease

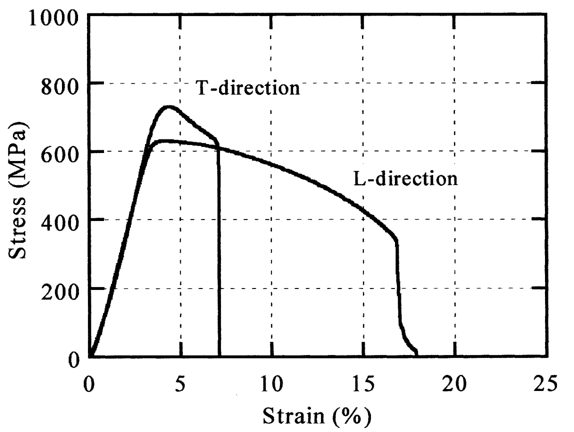

Fig. 1. Stress vs. strain curves of T-direction and L-direction tensile tests of cold worked plate.

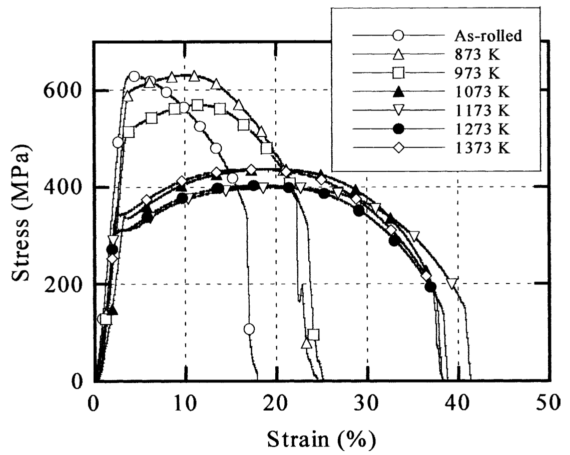

Fig. 2. Change in stress vs. strain curves of materials heat treated at different temperatures.

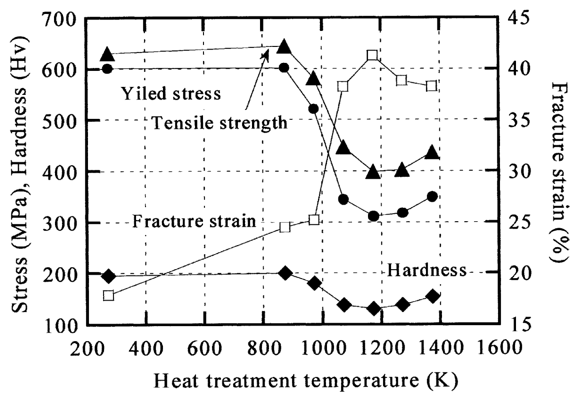

Fig. 3. Changes in yield stress, tensile strength, fracture strain and hardness of cold worked material against heat treatment temperature.

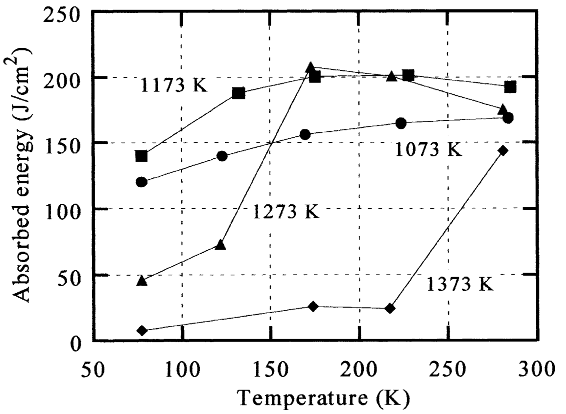

Fig. 4. Charpy test results against test temperatures. (Cold worked and annealed specimen. Temperatures in figure show annealing temperature.)

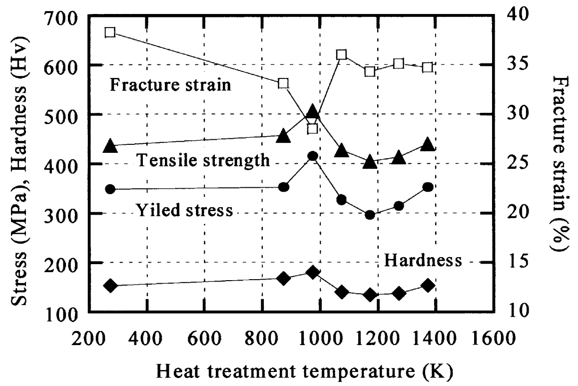

Fig. 5. Changes in yield stress, tensile strength, fracture strain and hardness of solution heat treated material against reheating temperature.

in absorbed energy against the test temperature. However, specimens annealed at 1273 and 1373 K display a ductile-to-brittle transition, with the higher transition temperature associated with the 1373 K annealing temperature. These results suggest that the matrix must become brittle as the annealing temperature goes up. As described later, the precipitation and solution behavior is responsible for this phenomena.

From these test results, a heat treatment temperature of 1073 K would be the best to recover the anisotropic deformation behavior and to retain a rather high yield stress and, moreover, to avoid the brittleness due to the solution hardening of the matrix.

The heavily rolled plate contains a lot of dislocations in the matrix and has an anisotropic microstructure. These are not suitable for microstructural investigation. Therefore, a solution heat treatment was performed to clean up the matrix and then the effect of subsequent heat treatments on impurity precipitation or dissolution was explored. The as-rolled plate was solution heat treated at 1373 K to recrystallize the microstructure, then reheating was performed to precipitate or dissolve the impurities. The heat treatments were conducted for 1 h in vacuum.

Fig. 5 summarizes tensile and Vicker's hardness test results as a function of heat treatment temperature for solution heat treated material. The hardness, yield and tensile strength peak at 973 K. On the other hand, the fracture strain is a minimum. When the annealing temperature increases to 1173 K, the hardness, yield and tensile strength are at minimum values. Heat treatment at 1373 K gives some recovery of hardness and tensile properties. The fracture strain is constant at around 35% and there is no clear difference over 1073 K. Fig. 6 shows the TEM observation results. There is no precipitation at 873 K, but very fine precipitates appeared at 1073 K. These precipitates would strengthen the matrix and give higher strength and hardness. It is a form of precipitation hardening. When the temperature increased to 1173 and 1273 K, the density of the precipitations decreased

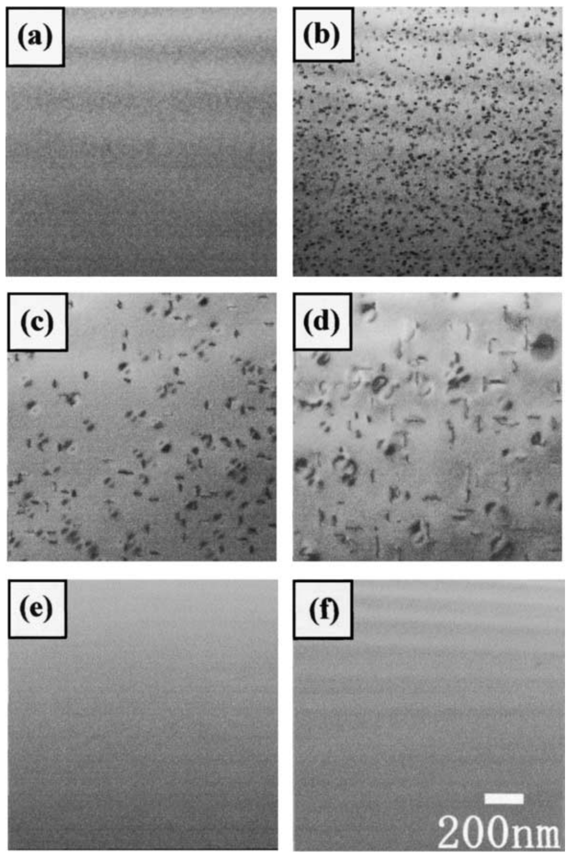

Fig. 6. TEM observation results of solution heat treated samples which were heat treated again at each temperature for 1 h. (a) 873 K. (b) 973 K. (c) 1073 K. (d) 1173 K. (e) 1273 K. (f) 1373 K.

and the precipitate size increased. These precipitates were analyzed and confirmed to be Ti–C–O. The concentration of these impurities in the matrix decreases, thereby softening the matrix. The heat treatments at 1273 and 1373 K do not cause precipitation and the hardness, yield and tensile strength all increase. The C, O, N impurities are dissolved in the matrix and they solution harden the material. From Fig. 4, it is noted that the matrix is embrittled when C, O, N are dissolved.

4. Conclusion

In this study, the mechanical properties were investigated in connection with the precipitation and solution behavior by changing the heat treatment temperatures. The main results are summarized as follows.

1. As-rolled specimens showed anisotropic deformation behavior and no strain hardening. Following heat treatment at and over 1073 K for 1 h, the work hardening due to rolling was recovered almost perfectly. Heat treatment over 1173 K did not generate further change in mechanical properties. The Charpy test revealed that 1273 and 1373 K heat treatments increased the DBTT significantly. This may be due to dissolution of Ti–C–O precipitates into the matrix.

2. Reannealing of solution heat treated specimens showed that heat treatment at 973 K after the solution heat treatment generated very fine precipitates of Ti–C–O in the matrix and increased yield stress and decreased elongation. Heat treatment at 1173 K softened the matrix, lowering the yield stress and increasing elongation. At temperatures over 1273 K, the precipitates were dissolved in the matrix producing brittle behavior due to solution hardening.

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