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Fabrication using electron beam melting of a V–4Cr–4Ti alloy and its thermo-mechanical strengthening study

H.Y. Fu [a], J.M. Chen [a,*], P.F. Zheng [a,b], T. Nagasaka [b], T. Muroga [b], Z.D. Li [c], S. Cui [c], Z.Y. Xu [a]

[a] Southwestern Institute of Physics, Chengdu, Sichuan, China
[b] National Institute for Fusion Science, Oroshi-cho, Toki, Japan
[c] General Research Institute for Nonferrous Metals, Xinjiehouwai 2, Beijing, China

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ABSTRACT

A 30 kg V–4Cr–4Ti ingot was fabricated by double electron beam melting for a structural materials application study for fusion reactors. The fabricated alloy was forged, hot rolled, and cold rolled to sheets of more than 90% deformation. Preliminary thermo-mechanical strengthening studies were completed, such as SACWA (solution annealing, cold working and aging), SAACW (solution annealing, aging and cold working), and SACWACWA (solution annealing, cold working, aging, cold working and aging), which were evaluated by hardness and tensile tests. Compared with traditional SAA (solution annealing and aging), multiplex strengthening is prominent via the imposed thermo-mechanical treatments, especially SACWACWA. SEM (scanning electron microscope) observation of the fracture surfaces was made after tensile testing; and all show ductile dimple fracture characteristics. In all conditions, there are coarse Ti-oxycarbonitride precipitates of about 200 nm in the grains or on grain boundaries. In the SA (solution annealing) and SAA conditions, there are just a few of the coarse precipitates on the boundaries, but in SAACW, the precipitates on the boundaries have a higher density and nearly contiguous morphology. This may be harmful for mechanical properties, especially in high-temperature environments, as these large precipitates may coarsen. There are also just a few coarse precipitates in the SACWA and SACWACWA conditions, while the density of nanometer-sized precipitates increased greatly, especially with SACWACWA. Cold working before aging manifests a better strengthening effect. This is possibly due to the dislocations, created in the cold working being pinned by precipitates formed in the succeeding aging process.

1. Introduction

Vanadium alloys, especially V–4Cr–4Ti alloys, have been considered as candidate structural materials for the first-wall and blanket components in future fusion reactors because of their low activation, excellent thermal stress factor, high-temperature strength, superior ductility at low temperatures, and high resistance to neutron irradiation [1,2]. Countries such as the USA and Japan have developed several hundred kilograms or tons of V–4Vr–4Ti alloys in the past [3,4]. Studies of vanadium alloys in China have remained at the laboratory scale in the past few years [5]. It is necessary to develop large-scale ingots, which is essential to establish the fabrication technology for components of blanket systems [4]. A 30 kg V–4Cr–4Ti alloy named SWIP-30, with a sum total of C, N and O of about 400 wppm, was fabricated by electron beam melting. In this study, the technology for fabrication of SWIP-30 has been demonstrated, which made it possible to investigate quantity production of vanadium alloys.

Several thermo-mechanical strengthening effects were also studied for further strengthening. Inducing high densities of precipitates and dislocations in the matrix is helpful to enhance the high temperature strength of V–4Cr–4Ti alloys [6]. The present authors have applied two-step heat treatments to V–4Cr–4Ti alloys to introduce high densities of small precipitates followed by cold working. Cold working can retard precipitate coarsening during aging [7]. The purpose of the current experiments is, evaluation of the effect of thermo-mechanical treatment (TMT) method with different combinations of CW (cold working) and aging (A), especially repeated CWA, on the strengthening of this vanadium alloy.

2. Fabrication of V–4Cr–4Ti plates by electron beam melting

The fabrication process of the V–4Cr–4Ti plates is shown in Fig. 1. The raw materials consisting of V, Cr and Ti powders pure more than 99.95% are about several millimeters in diameter. Before electron beam melting, the raw materials were pressed into an electrode with homogeneous distribution, proper density and good conductivity. Then the electrode was vacuum degassed to reduce gaseous impurities. Electron beam melting was performed in

* Corresponding author. Tel.: +86 28 82850415; fax: +86 28 82850956.
E-mail address: chenjm@swip.ac.cn (J.M. Chen).

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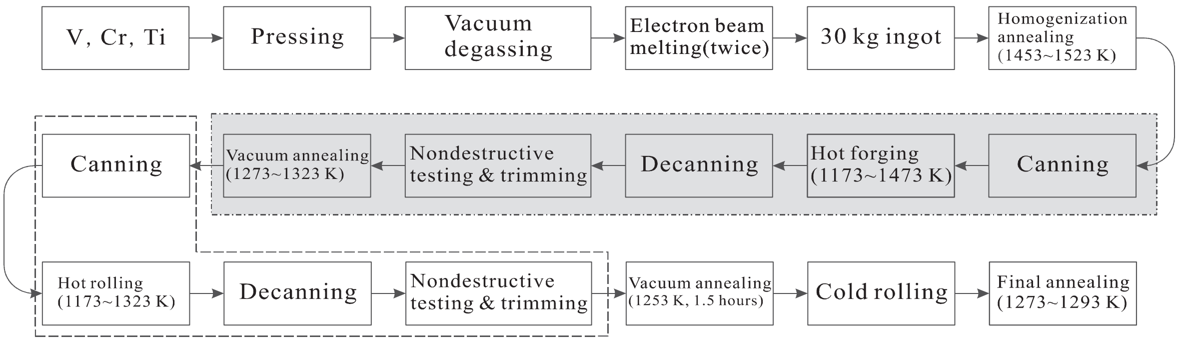

Fig. 1. Fabrication process of V–4Cr–4Ti plates.

vacuum at pressures less than 5×10^{-3} Pa. For homogeneity, the ingot was remelted. Finally, a 30 kg V–4Cr–4Ti ingot was prepared. While in the process of melting, segregation may occur and high casting stresses are anticipated. The electron-beam remelted ingot was annealed at 1453–1523 K in vacuum at pressures less than 3×10^{-2} Pa to improve homogeneity and machinability. The ingot was then canned with stainless steel to reduce air contamination during the hot-working processes, which induced both hot forging and hot rolling. Hot forging was conducted at 1173–1473 K with multi-step forming; after each pass, decanning, nondestructive testing, trimming and intermediate annealing were carried out. During hot forging, these procedures were implemented repeatedly until 50% deformation was achieved. In a similar way, during hot rolling, the alloy was canned. After more than 5 rolling passes, about 85% deformation was obtained, which was calculated from the hot-forged plates that were about 6 mm thick. After hot rolling, the plates were annealed below 10^{-3} Pa. The final process was cold rolling to 5 mm thickness and final vacuum annealing at 1273–1293 K. The chemical compositions of the final plates are shown in Table 1. For further deployment of vanadium alloys in advanced fusion systems, it is necessary to minimize Nb (20 wppm), Mo (50 wppm) and Ag (1 wppm) for neutron activation; Si must be optimized (400–1000 wppm) to suppress neutron-induced swelling and other minor impurities (O (400 wppm), N (200 wppm), S (30 wppm), and P (30 wppm), etc.) must be controlled to avoid grain boundary segregation and embrittling phases [3]. SWIP-30 almost satisfies these chemical composition requirements.

3. Thermo-mechanical strengthening study

3.1. Experiment

The following preliminary study of thermo-mechanical strengthening was completed in collaboration with the National Institute for Science, Japan. The condition of the as-received V–4Cr–4Ti (SWIP-30) was 5 mm plates, which were cold rolled and vacuum annealed at 1273 K for 2 h. The material was then cold worked for different deformation ($\geqslant$90%) levels for different combinations of heat treatment and CW (cold working) study. Table 2 shows the details of the experiment. The CW deformation change is listed in the table. All the heat treatment experiments have been done in vacuum at pressures less than 5.22×10^{-4} Pa to avoid additional interstitial impurity contamination (such as oxygen) from the atmosphere. The parameters of the STD (standard annealing), SA (solution annealing) and A (aging) heat treatment were 1273 K for 2 h, 1373 K for 1 h and 873 K for 20 h, respectively. Hardness and tensile tests have been done for mechanical property evaluations. Miniature samples with a gauge size of $0.25 \times 1.2 \times 5$ mm^3 were used for tensile and creep tests. Hardness test was performed using 300 g/30 s, and the strain rate of the tensile test both at RT (room temperature) and HT (high temperature) was 6.7×10^{-4} s^{-1}. High-temperature tensile tests also were performed in vacuum at pressures less than 5.22×10^{-4} Pa. Creep tests were carried out at 1073 K/170 MPa. Scanning electron microscopy was utilized for fracture surface and microstructure observations.

3.2. Results and discussion

Fig. 2 shows the grain structure of SWIP-30 treated at different conditions, separately. After solution annealing at 1373 K for 1 h (SA), the grain structure is equiaxed with size of about 30 μm. When solution annealed and then aged (SAA), the grains are also equiaxed in shape. But when cold worked, such as SAACW, SACWA, and SACWACWA, the grains were elongated with aspect ratios of about 2.23, 1.757 and 2.27, respectively.

Hardness comparisons of SWIP-30 at different conditions are shown in Fig. 3. Compared with STD and SAA, when a thermo-mechanical treatment is applied, such as SAACW, SACWA, SACWACWA, the hardness is greatly increased, especially for SAACW and SACWACWA, which have a hardness of 246 HV (2.413 GPa).

Fig. 4 shows tensile results of SWIP-30 at different conditions. The results show that, when tensile tested at RT, SAACW has the highest strength but the smallest elongation; SACWACWA has the second highest strength. When tested at 973 K, SAACW has the same strength as that of SACWACWA, and the elongation is a little larger than the latter. SACWA also has good strength and elongation. When tested at 1073 K, SACWACWA has better strength and ductility than that of SACWA.

Table 1
Chemical composition of main elements and impurities (wt.%).

Cr	Ti	C	N	O	S
3.81	3.92	0.013	0.0020	0.027	0.0020
Al	Si	K	Fe	Mg	Ca
0.010	0.059	<0.005	0.0053	0.0022	0.0067
Ge	Mo	Na	Ta	Zr	Ni
<0.001	0.0035	<0.005	<0.001	<0.001	0.0082

Table 2
Parameters of thermo-mechanical strengthening study.

Series	Abbreviation	Deformation calculated from the as-received	Tensile test at
#1	STD	95%	RT
#2	SAA	95%	RT, 700 °C, 800 °C
#3	SAACW	94%, and then 17%	RT, 700 °C
#4	SACWA	94%, and then 17%	RT, 700 °C, 800 °C
#5	SACWACWA	90–40%, and then to 17%	RT, 700 °C, 800 °C