MICROSTRUCTURAL CHARACTERISTICS AND MECHANISM OF TOUGHNESS IMPROVEMENT OF LASER AND ELECTRON-BEAM WELDS OF V–4Cr–4Ti FOLLOWING POSTWELDING HEAT–TREATMENT*

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SUMMARY

We are conducting a program to develop an optimal laser welding procedure for large–scale fusion–reactor structural components to be fabricated from vanadium–base alloys. Microstructural characteristics were investigated by optical microscopy, X–ray diffraction, transmission electron microscopy, and chemical analysis to provide an understanding of the mechanism of the drastic improvement of impact toughness of laser and electron-beam (EB) welds of V–4Cr–4Ti following postwelding annealing at 1000°C. Transmision electron microscopy (TEM) revealed that annealed weld zones were characterized by extensive networks of fine V(C,O,N) precipitates, which appear to clean away O, C, and N from grain matrices. This process is accompanied by simultaneous annealing-out of the dense dislocations present in the weld fusion zone. It seems possible to produce high–quality welds under practical conditions by controlling and adjusting the cooling rate of the weld zone by some innovative method to maximize the precipitation of V(C,O,N).

INTRODUCTION

For the vanadium alloys, recent attention has focused on development of welding procedures and investigation of the weld properties of the reference alloy V–4Cr–4Ti. The objective of this task is to develop an optimal laser welding procedure that can be applied to welding of large–scale fusion–reactor structural components. Initial investigation of mechanical properties of laser and EB welds of the production–scale heat of V–4Cr–4Ti (500–kg Heat #832665) has demonstrated that impact toughness of the welds is improved drastically by postwelding heat–treatment (PWHT) at ≈1000°C for 1 h.[1]

Although postwelding annealing at 1000°C for as long as 1 h is obviously impractical for field application, it was thought to be important to understand the mechanism of the drastic improvement of the impact toughness, because once the mechanism is understood, it would be possible to develop an innovative method to apply the underlying principle to produce a good weld under practical conditions. Therefore, a microstructural investigation by optical microscopy, X–ray diffraction, TEM, and chemical analysis has been conducted on as-welded and postwelding-annealed specimens of V-4Cr-4Ti to provide an understanding of the mechanism of the toughness improvement.

MICROSTRUCTURAL ANALYSES

Optical Microscopy

Optical micrographs of the as-welded and postwelding-annealed specimens of the laser weld are shown in Fig. 1. The low- and high-magnification photomicrographs in the figures were obtained from the side of the broken or bent Charpy specimens that were impact-tested after the heat treatment. Elongated grains and fracture line in the weld fusion zone are visible in Fig. 1(A), the low-magnification phototmicrograph.

In the high-magnification photomicrograph of the postwelding-annealed material, Fig. 1(C), a characteristic fine substructure is visible within the elongated grains of the fusion zone. This substructure is absent in the

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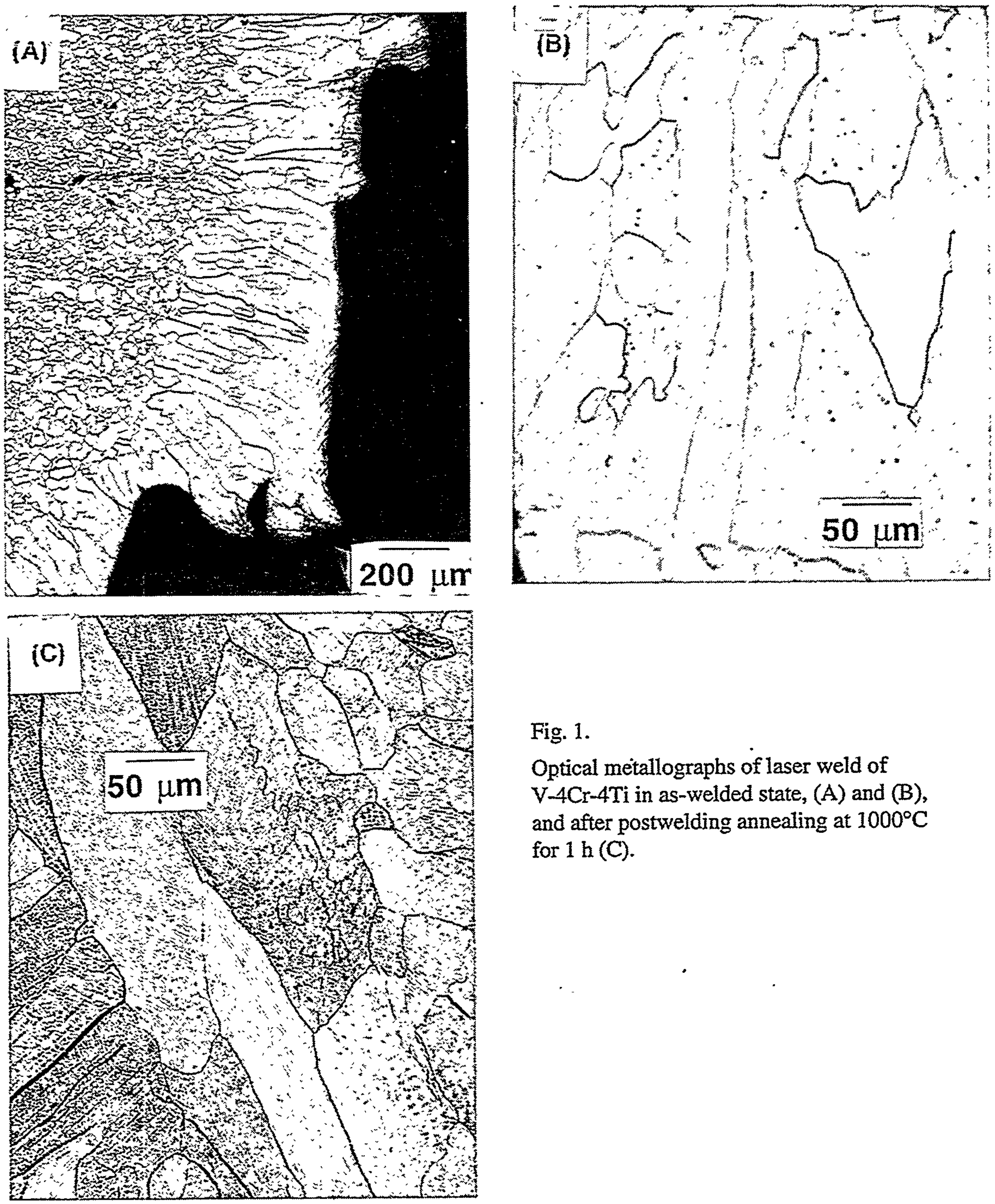


Fig. 1.

Optical metallographs of laser weld of V-4Cr-4Ti in as-welded state, (A) and (B), and after postwelding annealing at 1000°C for 1 h (C).

as-welded material, Fig. 1(B). In EB welds, early-stage development of the similar fine substructure was observed even in as-welded material, Fig. 2(B). In the postwelding-annealed material, the substructure appears to have developed into an advanced stage, Fig. 2(C). Thus, identification of this fine substructure seems to be the key to understanding the mechanism of toughness improvement following the PWHT.

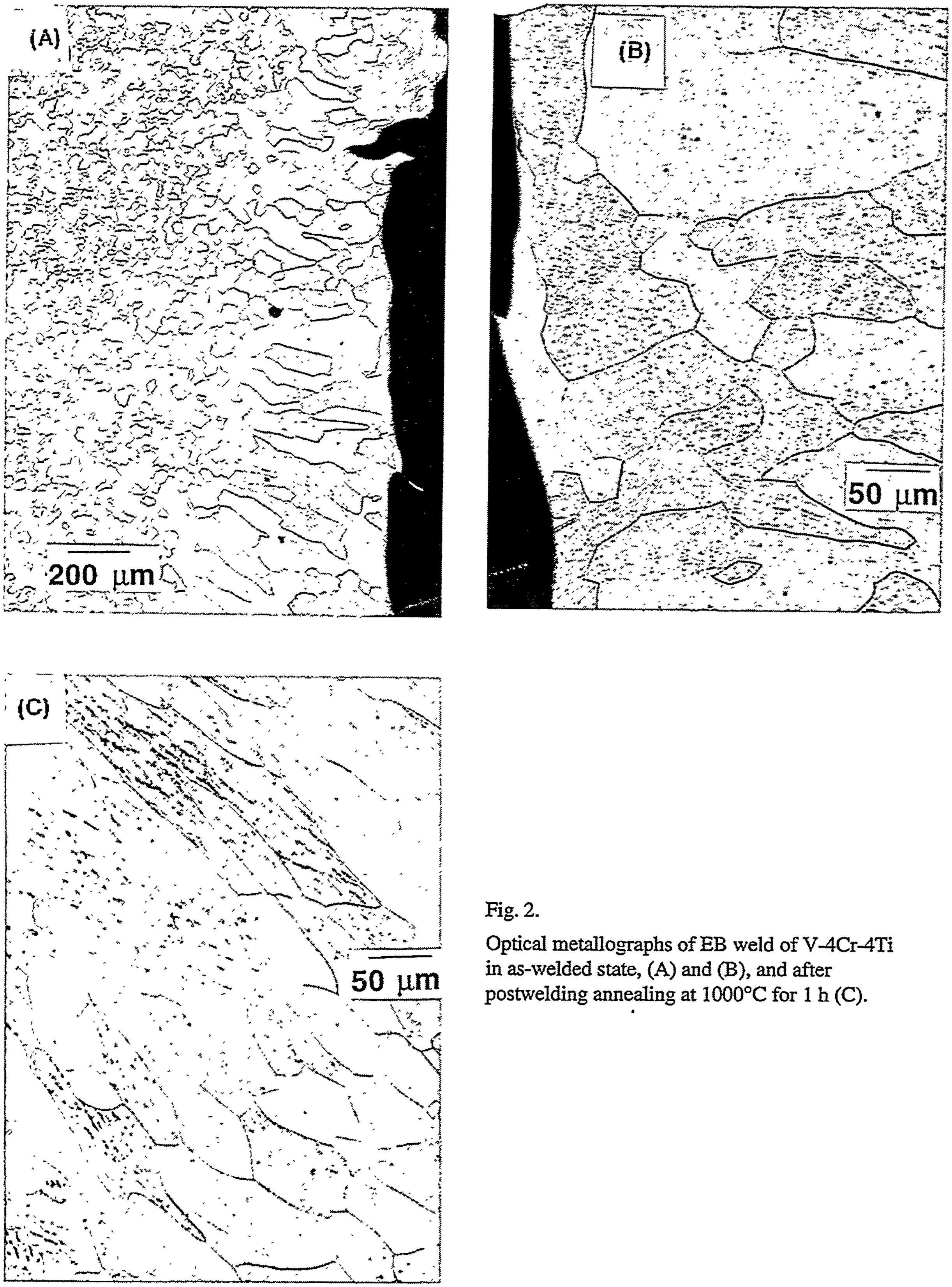


Fig. 2.

Optical metallographs of EB weld of V-4Cr-4Ti in as-welded state, (A) and (B), and after postwelding annealing at 1000°C for 1 h (C).

Chemical Analysis

Concentrations of O, C, and N were analyzed in EB welds. Three specimens of base metal and three specimens of EB weld were analyzed. These concentrations were compared in Table 1 with those measured on an extruded plate (64 mm thick) and a rolled base-metal plate (3.8 mm thick). The base-metal plate was annealed in the factory at ≈1050°C for 2 h and subsequently used in the present welding. Compared to the composition of the rolled and annealed base–metal plate, increase in O, N, and C in the EB welds was insignificant. Therefore, contamination by O, N, and C in the EB and laser welds appears to be at best a secondary factor in the large shifts in the ductile-brittle transition temperature (DBTT) before and after welding.[1]

Table 1. Impurity concentration (in wppm) in 500-kg V-4Cr-4Ti Heat #832665 after extrusion, rolling and annealing, and electron-beam welding

	O		N		C	
Material	reading	average	reading	average	reading	average
Extruded plate	310	310	85	85	80	80
Rolled Plate	450, 480, 467	466	25, 28, 30	27	300, 230, 240	257
EB weld	510, 520, 520	517	30, 25, 29	28	240, 270, 240	250

X-Ray Diffraction Analysis

Tetragonal distortion was suspected initially in the crystallographic structure of the elongated grains in the fast-cooled weld fusion zone, which was thought to be associated with the high DBTT. However, X-ray diffraction analysis showed no evidence of tetragonal distortion; only the diffraction peaks that correspond to the bcc structure of vanadium were present, i.e., (110), (200), (211), (220), and (310). However, the lattice constant of the weld fusion zone was found to be ≈0.13% larger than that of the base metal, i.e., 0.30315 vs. 0.30275 nm, respectively. These lattice constants were used to index the TEM diffraction patterns (see below).

TEM Analysis

As-welded microstructures of laser and EB welds were characterized by dense dislocations as shown in Fig. 3. Ti(O,N,C) precipitates normally present in extruded, rolled, and annealed plates were conspicuously absent, showing that they had dissolved in the grain matrices during welding. Reprecipitation of Ti(O,N,C) during cool-down of the weld fusion zone was negligible.

Postwelding-annealed laser and EB welds were characterized by an extensive network of precipitates that were not observed in the base metals of any vanadium alloys investigated in this program. Examples of the network precipitate structures are shown in Figs. 4 and 5, for laser and EB welds, respectively. Individual precipitates were typically rodlike in shape, 200-500 nm in length, and ≈50-100 nm in diameter. The rodlike morphology indicates immediately that the precipitates are not Ti(O,N,C), which is usually spherical or ellipsoidal and 300-500 nm in diameter. Dark-field morphologies shown in Figs. 3(B) and 4(B) indicate that the precipitates in the laser and EB welds are of the same type.

To obtain a clue as to the nature of the precipitates, selected precipitates were analyzed by energy-dispersive spectroscopy (EDS). A few precipitates located at the edge of the of the hole of the perforated TEM foil were analyzed. The measured EDS spectrum, therefore, consisted of X-rays that originated predominantly from the precipitate, while X-rays from the alloy matrix were negligible. Results of the EDS analysis of the precipitate and alloy matrix, given in Table 2, show that the precipitate is a vanadium-base phase rich in C and O.

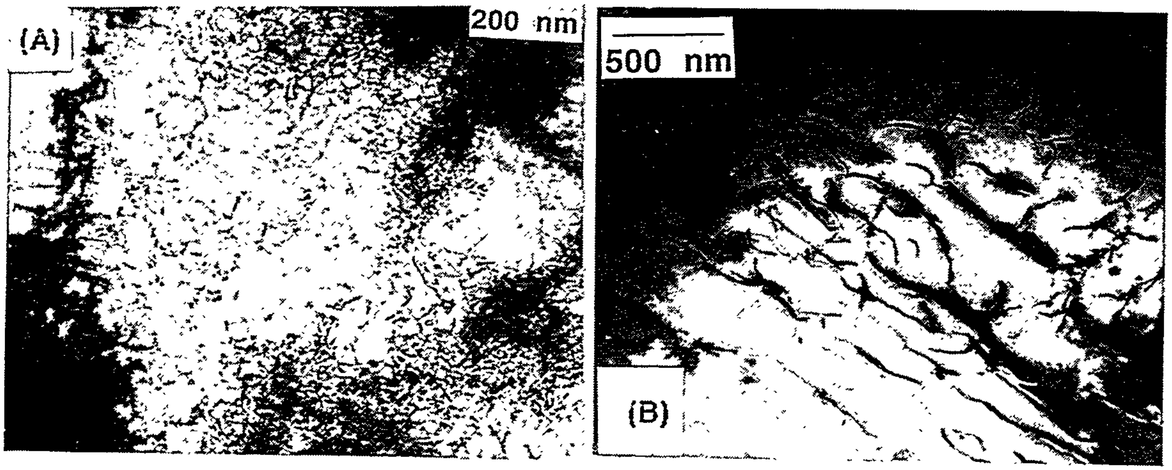


Fig. 3 TEM bright-field micrographs of laser (A) and EB (B) welds of V-4Cr-4Ti before post-weld anneling. Note dense dislocations and absence of the normal Ti (O, N, C) precipitates.

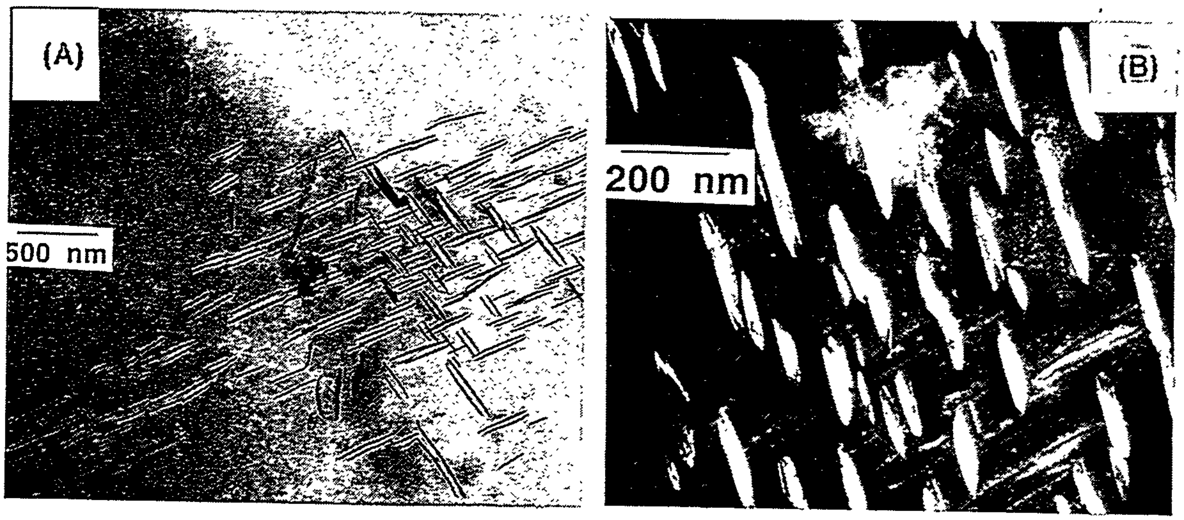
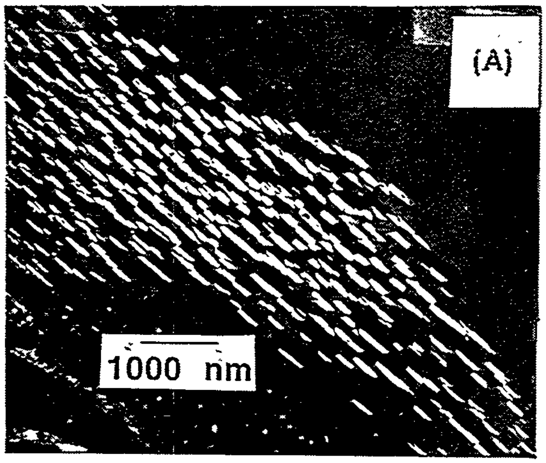


Fig. 4. Bright- (A) and dark-field (B) photomicrographs of laser weld fusion zone of V–4Cr–4Ti after postwelding annealing at 1000°C for 1 h. Note dense precipitate substructure and absence of dislocations and Ti(O,N,C).

Table 2. Summary of EDS analysis of the composition (at.%) of the characteristic precipitates in the laser weld fusion zone after postwelding annealing at 1000°C for 1 h in vacuum

	V	Ti	Cr	C	O
Matrix	92.55	3.68	3.77	-	-
Precipitate	16.72	5.94	0.76	66.99	9.59



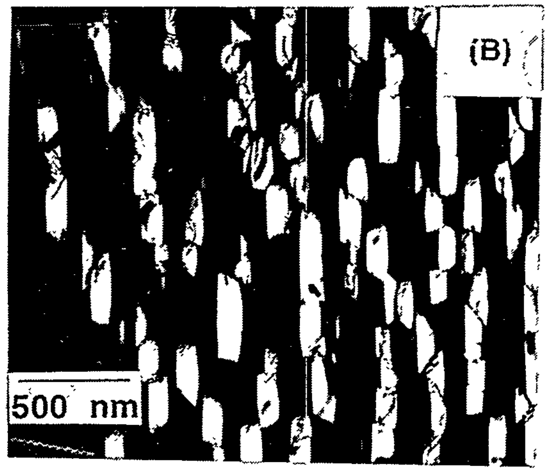


Fig. 5. Dark-field (B) micrographs of precipitates in EB weld fusion zone of V-4Cr-4Ti after postwelding annealing at 1000°C for 1 h.

However, exact compositions of C and O in this type of analysis must be considered as only qualitative, because contamination of C from the TEM vacuum chamber and hydrocarbon thinning solution is possible. Furthermore, accurate determination of C and O by EDS is complicated because the weak K lines of C and O nearly overlap the L lines of V and Ti. Therefore, exact identification of the precipitate phase must be verified by a more precise analysis through dark–field imaging and indexing selected-area diffraction patterns.

Systematic analysis by dark-field imaging and selected-area-diffraction (SAD) showed that the characteristic precipitates are V(C, O, N), which is an fcc phase with a lattice constant of 0.419 nm. An example of the indexed diffraction patterns is shown in Fig. 6. In this pattern, one zone axis of vanadium and two zone axes of V(C, O, N) phase are operating. Indexed diffraction patterns obtained from the laser and EB welds showed that the precipitates formed in the two types of welds are the same V(C,O,N) phase.

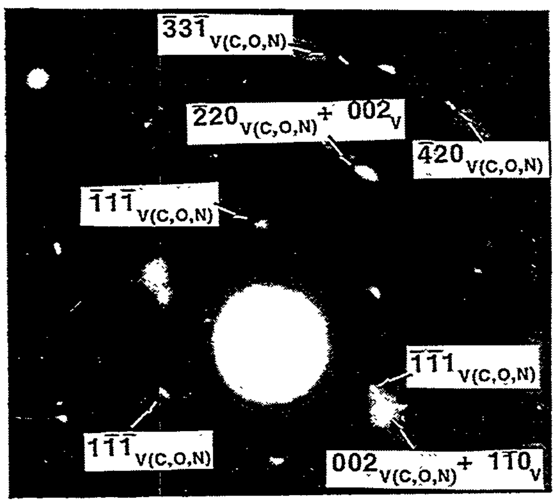


Fig. 6.

Indexed selected area diffraction pattern of laser weld fusion zone of V~4Cr–4Ti postwelding-annealed at 1000°C for 1 h.

DISCUSSION

VC (lattice constant 0.417 nm), VO (0.409 nm), and VN (0.413 nm) phases are isostructural (fcc, Na–Cl type) and have similar lattice constants[2] and high miscibility with one another.[3] This is similar to the characteristics of TiC, TiO, and TiN, which are also isostructural fcc with similar lattice constants and high miscibility. Therefore, as for Ti(C,O,N), the vanadium-base precipitates characteristically contained in the postwelding-annealed welds are believed to be in the chemical form of V(C,O,N), with variable proportions of C, O, and N in the precipitate. For the precipitate analyzed in Table 2, N content was negligible. The substructures observed in the low-magnification optical photomicrographs of Figs. 1 and 2 are indeed the same as the precipitate network shown in the high-magnification TEM photomicrographs of Figs. 4 and 5.

Vanadium-base precipitates have been observed only rarely in V-Ti or V-Cr-Ti alloys.[4–6] In the Ti-containing binary or ternary alloys, observed precipitates are usually Ti-based phases, such as titanium oxycarbonitrides, titanium sulfides, or titanium phosphides.[4] Vanadium–base precipitates were observed only in alloys containing high levels of unusual impurities such as Cl, Ca, and Li, i.e., vanadium oxychlorides in V-5Cr-5Ti, which was melted with low-quality sponge Ti[5] and Ca-vanadate in unalloyed vanadium produced by the calcia-reduction process.[5] In an irradiated V-20Ti alloy rich in boron, Li-vanadate was also observed.[6]

As pointed out previously, from the thermodynamic standpoint, the precipitation of vanmadium oxychlorides or Ca-vanadates seems to be preferred over precipitation of Ti-based precipitates in Cl- or Ca-rich alloys containing a certain level of oxygen. Precipitation of V(C,O,N) seems to be preferred over precipitation of Ti(C,O,N) in a metastable structure such as the weld fusion zone. A laser or EB weld fusion zone contains dense dislocations and higher levels of O, N, and C as interstitial solutes in the grain matrices following dissolution of Ti(O,N,C) during melting. It appears that a dense dislocation structure plays an essential role in the precipitation of V(C,O,N) in the welds. The network-like distribution of clusters of the V(C,O,N) precipitates shown in Figs. 4 and 5 seems to support this premise.

Under the same annealing condition at 1000°C for 1 h in high vacuum, V(C,O,N) precipitates were not observed in the factory-annealed base metal, which is relatively free of dislocations and contains the normal Ti(O,N,C) precipitates. This seems to be additional evidence that high-density dislocations play an important role in the precipitation of (V(C,N,O) in the welds during postwelding-annealing.

It seems evident that the drastic improvement in impact toughness is a result of the simultaneous process of profuse formation of V(C,O,N) precipitates and annealing-out of the dense dislocations that occurs in the weld zone during the postwelding annealing. The combined process seems to make grain matrices that are very low in O, C, and N and virtually free of dislocations and residual stress. Crack propagation through this type of microstructure would then be very difficult, which leads to excellent impact toughness.

The precipitation kinetics of V(C,O,N) in the metastable structure of laser welds are believed to be strongly influenced by annealing temperature. Therefore, identification of the temperature of fastest precipitation kinetics in the time-temperature-transformation (TTT) curve will be important. This temperature is probably significantly higher than 1000°C, and the kinetics at that temperature seem to be fast. This can be deduced from the observation of the early-stage development of the precipitate network in EB welds even without postwelding annealing, Fig. 2(B). A controlled cooling of a laser weld would then be an attractive idea, in which the weld structure remains at the temperature of maximum precipitation kinetics for a reasonable period of time under reasonably practical conditions.

CONCLUSIONS

1. Postwelding–annealed welds were characterized by extensive formation of networks of fine V(C,O,N) precipitates. This process occurs with simultaneous annealing-out of the dense dislocations present in the metastable weld fusion.

2. The drastic improvement in impact toughness is a result of this simultaneous process, which occurs in the weld fusion zone during the postwelding annealing at 1000°C for 1 h. The combined process seems to make grain matrices that are very low in O, C, and N and virtutally free of dislocations and residual stress. Resistance to crack propagation through the grains of this type of microstructure seems to be high, and as a result, excellent impact toughness is produced.

3. The precipitation kinetics of V(C,O,N) in the metastable structure of laser welds are predicted to be strongly influenced by annealing temperature, and hence by cooling history. Therefore, it seems possible to produce high–quality welds under practical conditions by controlling and adjusting the cooling rate of the weld fusion zone through some innovative method to maximize the precipitation of V(C,O,N).

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