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学 位 論 文 題 目　Impurity behavior and weld joint properties of low activation vanadium alloys for fusion reactors

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論文内容の要旨

Vanadium alloys have been regarded as one of the promising candidate structural materials for fusion first wall/blanket applications, due to their low induced radioactivity, good compatibility with lithium, good resistance to neutron radiation damage and good strength at elevated temperature. Some critical issues such as large heat fabrication technology, unirradiated and irradiated mechanical properties, chemical compatibility, and corrosion properties have been studied to apply the vanadium alloys for the structural components of fusion reactors. Based on the previous studies, V-4Cr-4Ti alloy was selected as a leading candidate.

One of the key issues for the vanadium alloys is the welding process, which is the requisite for fabricating structures. It is important to control the environment during the welding for preventing the contamination with the impurities such as oxygen, nitrogen and carbon, because it is well known that those impurities have various effects on the properties of vanadium alloys. Some research efforts for the welding on vanadium alloys have been carried out with gas-tungsten arc, electron beam, laser and so on. Laser welding has some unique characteristics when compared to other welding processes. It does not require either a complete electrical circuit, or a vacuum chamber or shielding for X-ray. It should be noted that the blankets for fusion reactor are mainly composed of thin plates, their morphology is very complex and it is necessary to repair them by remote control. Thus, the YAG laser welding is a quite attractive technique for application to the fusion blanket because of its flexibility for field, lenient requirement for atmospheric control, capability of deep penetration with less input, and remote operation capability. Some researches for the laser welding on vanadium alloys have been carried out, but most of them failed to prevent the contamination with the impurities, resulting in the degradation on the properties of the weldments.

The purpose of this study is to develop a laser welding technology for low activation V-4Cr-4Ti alloys. For this purpose, an environmental control box was fabricated to eliminate the contamination with the interstitial impurities, and then the YAG laser welding was carried out inside it. The effect of the welding conditions on the mechanical properties of the weld zone, such as hardness, bending, tensile and Charpy impact properties, and the impurity behavior in the weld zone by microstructural observation for the mechanistic understanding of the property change, based on the understanding of hardness and precipitation behavior by heat treatment were investigated.

As the basic study supporting the development of the welding technology, it is crucial to enhance the understanding on the impurity behavior under various thermomechanical treatment processes. In the present study, the effect of impurity levels on hardness and precipitation behavior by various heat treatments was also investigated. The previous studies on the impurity effects on vanadium alloys revealed that even if the initial impurity content is rather high, good properties can be acquired by optimal heat treatment resulting in the formation of the precipitates with the impurities. Thus, the key issues for fabrication of vanadium alloys are to eliminate the pickup of the critical impurities from the atmosphere during the fabrication and working process and to determine the optimal heat treatment for vanadium alloys. Therefore, this study will be also valuable for optimizing the fabrication and thermomechanical treatment conditions of the alloys, as well as for the welding development.

To investigate the impurity behaviors by heat treatment, V-4Cr-4Ti model alloys doped with various levels of oxygen and nitrogen were fabricated by button arc melting. Model alloys were machined into 10 mm thick slabs from the buttons. All the sheets were cold-rolled to a reduction of > 50% in thickness, followed by annealing at 1373 K for an hour in a vacuum ($<10^{-3}$ Pa). The specimens were then heat-treated again from 873 to 1373 K for an hour. Vickers hardness testing and microstructure observation were carried out after annealing.

Impurities existed in both the solid solution and the precipitates. Two kinds of precipitates were observed in the specimens doped with oxygen: large and small precipitates. They were identified as Ti-rich cubic phases and Ti-C-O compounds, respectively. In the specimens doped with nitrogen, however, only the large precipitates were observed. The large precipitates were stable in all heat treatments to 1373 K, but the fine precipitates appeared at 973 K and vanished at 1373 K. The density of the large precipitates increased with nitrogen level more than oxygen level. The density of the fine precipitates increased with oxygen level. Most of the nitrogen impurities are included into the large precipitates and are stable during the heat treatments to 1373 K. Some fraction of the oxygen impurities will be in large and fine precipitates. The formation of the fine precipitates at 973 K resulted in the increase in hardness by the precipitation hardening. The hardness then decreased with the increase in annealing temperature due to the decrease in the precipitates density. The oxygen in the fine precipitates will be released into matrix by annealing at 1373 K by resolution of the precipitates, resulting in another increase in hardness by the solid solution hardening.

According to the present results, the annealing temperature will be optimized at 1273 K or below, taking the precipitation hardening and the solid solution hardening into account. Heat treatment at above 1273 K will result in the degradation of the properties.

In the welding experiments, high-purity V-4Cr-4Ti alloy (NIFS-HEAT-2) fabricated by National Institute for Fusion Science was used. The specimen was prepared by annealing at 1273 K for 2 hours before laser welding. In this study, the laser welding technology has been developed for vanadium alloys eliminating the contamination. To prevent contamination with interstitial impurities, such as hydrogen, oxygen, nitrogen, carbon, etc. during welding, the laser welding was carried out in an environmental control box capable of supplying argon gas and ventilating the fume. Bead-on-plate welds were produced on the 4 mm-thick plate using 2.0 kW YAG laser. Chemical analysis of the weld metal was carried out to measure the contaminations. The microstructure was also observed with optical microscope (OM), scanning electron microscope (SEM) and transmission electron microscope (TEM).

The hardening occurred in the weld zone. This was quantitatively explained by the experiments on the impurity behaviors in the V-4Cr -4Ti alloys. Increase in hardness of the weld zone was due to the dissolution of large and small precipitates. In the weld metal, dissolution of both large and small precipitates resulted in remarkable hardening. Only small precipitates disappeared at a distance from the weld metal, resulting in smaller hardening.

Soundness of the weld joint was confirmed by estimating mechanical properties. In the tensile test of the joint, decrease in tensile strength did not occur but total elongation decreased slightly in the weld zone. The absorption energy of the weld metal produced by the low power density was as high as that of the base metal. Decrease in the absorption energy in the case of the high power density seems to be due to the increased width of the weld metal and the heat-affected zone (HAZ) and the increased grain size in the weld zone. To improve the absorption energy of the weld metal, it is effective to decrease the input power density for reducing the grain size and the width of the weld metal and the HAZ.

In summary, this study has demonstrated that the laser welding of vanadium alloys maintaining their good properties is possible by a simple environmental control. The basic understanding of the behavior of interstitial impurities such as oxygen, nitrogen and carbon, during various thermomechanical treatment derived in this study was applied to explaining the properties of the weldment and to improving the procedures of thermomechanical treatment and welding.

論文の審査結果の要旨

核融合エネルギーの実現に向けての重要な工学課題の一つに、構造材料の開発が挙げられる。核融合炉の構造材料は、長期間高温中性子照射に耐える必要があるとともに、使用後の効率的な廃棄や再利用の可能性を考えると、誘導放射能の減衰が早いことが求められ、材料の構成元素が著しく制限される。現在工業化されている材料ではこのような要件を満たすことは難しく、先進的な新材料を開発する必要があると考えられている。バナジウム合金は、低放射化特性や高温強度、燃料増殖材の候補である液体リチウムとの共存性など優れた特性を有する最も有望な候補材の一つであるが、工業基盤が殆ど無いため、製造加工法、溶接法など基盤技術の確立が必要である。バナジウム合金は活性金属であり、酸素、窒素不純物濃度やそれらを含む析出物の状態により強度特性が大きく変わることが知られている。従ってバナジウム合金の開発に向けては、熱加工過程や溶接過程における不純物挙動の基礎的理解が重要である。本研究は、低放射化バナジウム候補合金（V-4Cr-4Ti）の熱加工履歴による不純物挙動の基礎機構を明かにするとともに、それらの結果を溶接材における不純物挙動の評価に適用し、溶接法の高度化への指針を得ることを目的としたものである。

本研究では、V-4Cr-4Ti 共通材料に加え、酸素、窒素濃度を独立に変化させた様々なモデル合金を作製し、酸素、窒素それぞれについて析出物形成、再固溶挙動の熱履歴依存性およびその硬さ変化への影響を、様々な組織観察と強度特性試験により定量的に明らかにした。窒素が、溶解後熱間加工時に形成される析出物に殆ど吸収され、1100℃付近まで安定に存在すること、一方酸素は600℃付近で Ti と化合物を形成するが、1000℃以上で再溶解し、母相の固溶硬化を引き起こし強度特性が劣化することなど、従来狭い温度で部分的に調べられていた不純物効果について、広い温度範囲でしかも酸素と窒素を分離してその挙動を系統的に明らかにしたことは特筆すべき成果である。これらの結果から、バナジウム合金の最適な熱加工履歴を導き出すことができたことは、バナジウム合金の工業基盤の確立に貢献するものである。

本研究では、続いてこれらの基礎研究成果に基づいて、バナジウム合金の YAG レーザー溶接法の適用性について検討を進めた。溶接時の不純物混入が溶接部の特性を劣化させることが懸念されるので、不純物混入を防ぐチャンバーを自ら設計製作し、YAG レーザー溶接部の組織、強度特性とその溶接パラメータ依存性を詳細に明らかした。そして、モデル合金による不純物挙動の基礎データを用いて溶接部に起こる硬さ変化の微視的過程を明らかにするとともに、不純物挙動を制御する観点から、溶接法の高度化に向けた指針を得た。従来バナジウム合金の溶接試験では、雰囲気管理が不充分であり殆どの場合母材より溶接部の強度特性は劣化したが、本研究では厳密な不純物管理により母材と遜色のない溶接部材の製作が可能になった。このような制御された溶接試験に基づいて溶接部での不純物の挙動が詳細に検討されている。本研究により、母材の不純物濃度を下げること、析出物の再固溶効果と結晶粒の粗大化による特性劣化を防ぐため溶接入熱を抑えることなど、基礎機構の理解に基づいた溶接の高度化の具体的な指針が得られたことの意義は大きい。

本論文は、以上の研究を適切にまとめており、核融合炉用低放射化バナジウム合金に関して、不純物挙動の基礎研究に基づいて、製造、加工、接合プロセスの発展に貢献するも

のとして、実用的および学術的な面でともに価値が高い研究であると認められる。よって本審査委員会は、本論文が博士学位論文として充分な水準にあるとの結論に達した。