マテリアル先端リサーチインフラ利用報告書
ARIM User's Report

【Release：2024.07.25】　【Update：2024.03.28】

課題データ / Project Data

課題番号 Project Issue Number	23KU0025
利用課題名 Title	Ti合金における高温組織変化のその場観察
利用した実施機関 Support Institute	九州大学 / Kyushu Univ.
機関外・機関内の利用 External or Internal Use	外部利用/External Use
横断技術領域 Cross-Technology Area	計測・分析/Advanced Characterization
重要技術領域 Important Technology Area	革新的なエネルギー変換を可能とするマテリアル/Materials enabling innovative energy conversion 次世代ナノスケールマテリアル/Next-generation nanoscale materials
キーワード Keywords	金属材料,電子顕微鏡/ Electronic microscope,水素貯蔵/ Hydrogen storage,電子回折/ Electron diffraction

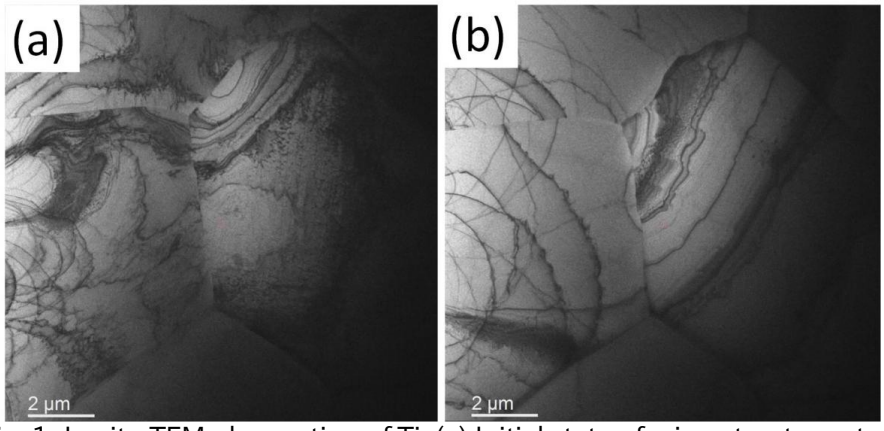
利用者と利用形態 / User and Support Type

利用者名（課題申請者） User Name (Project Applicant)	吉田 周平
所属名 Affiliation	京都大学大学院工学研究科
共同利用者氏名 Names of Collaborators in Other Institutes Than Hub and Spoke Institutes	Guo Baoqi
ARIM実施機関支援担当者 Names of Collaborators in The Hub and Spoke Institutes	前野 宏志
利用形態 Support Type	技術補助/Technical Assistance,機器利用/Equipment Utilization

利用した主な設備 / Equipment Used in This Project

利用した主な設備 Equipment ID & Name	KU-016：低温域観測型・高分解能電子顕微鏡

報告書データ / Report

概要（目的・用途・実施内容） Abstract (Aim, Use Applications and Contents)	Experimental studies on phase transformation from alpha-HCP phase to beta-BCC phase in Ti and its alloys is limited to ex-situ observations in which microstructure is observed at room temperature after rapid cooling. However, during cooling, beta phase transformed to martensite or alpha phase. Thus, it is very difficult to understand the mechanisms of real-time phase transformations though conventional ex-situ techniques. To solve this problem, we used transmission electron microscopy (TEM) with a heating specimen stage to study the phase transformation from alpha to beta in real-time.
実験 Experimental	Pure Ti was firstly heavily deformed by a high-pressure torsion process at room temperature and annealed at 800˚C for 3 min so that fine grains can be obtained. The samples with fine grains were introduced to TEM (ARM-300F2) with a heating specimen holder. The accelerated voltage was 300 kV. The sample was supposed to be heated to temperatures ranging from 900˚C to 950˚C with the heating rate of 0.5 ˚C / s. Diffraction contrast images of the microstructure and selective area diffraction patterns were continuously taken to observe the microstructural change during the phase transformation.
結果と考察 Results and Discussion	The initial microstructure prior to heating (Fig. 1(a)) showed that the four grains were detected with clear grain boundaries. After heating to 950 ˚C, grain boundary migration was detected (Fig. 2(b)). The bulging behavior of grain boundary was also observed. However, phase transformation was not detected in this sample. One reason is that geometrical factors such as sample thickness can affect the free energy balance of alpha and beta phases, resulting in increase in the transformation temperature (higher than the temperature limit of the facility). Another possible reason is that the temperature in the specimens is inhomogeneous so that the temperature of the observed area is lower than the target temperature, which is insufficient to activate the phase transformation from alpha to beta. Modifying chemical composition of the material may help us to activate the phase transformation, and it will be our future work.
図・表・数式 1 Figures, Tables and Equations 1	 Fig. 1: In-situ TEM observation of Ti. (a) Initial state of microstructure at room temperature and at (b) 950 degree C with holding for 120 s.
その他・特記事項（参考文献・謝辞等） Remarks(References and Acknowledgements)	This work was supported by JSPS KAKENHI Grant-in-Aid for Challenging Research (Exploratory) (JP22K18888). Also, the technical assistance of Mr. Hiroshi Maeno of Kyushu University is highly acknowledged.

成果発表・成果利用 / Publication and Patents

DOI（論文・プロシーディング） DOI (Publication and Proceedings)	

口頭発表、ポスター発表 および、その他の論文 Oral Presentations etc.	
特許出願件数 Number of Patent Applications	0件
特許登録件数 Number of Registered Patents	0件