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## THESIS OUTLINE

系・コース Department of, Graduate major in	Materials Science and Engineering Nuclear Engineering	系 コース	申請学位 (専攻分野): Academic Degree Requested	博士 Doctor of (Engineering)
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### Thesis Outline

English Title: Neutron Irradiation Effects on Defect Formation in Random and Highly Oriented Aluminum Nitride and Their Recovery Behavior by Thermal Annealing

Japanese Title: 無配向および高配向窒化アルミニウムの欠陥生成に及ぼす中性子照射の影響および熱アニールによる回復挙動

Aluminum nitride (AlN) ceramics have excellent properties such as high thermal conductivity, low thermal expansion, and low dielectric constant. They have been expected to be a promising material for nuclear applications as well as corrosion and magneto-hydrodynamic (MHD) barrier in future fusion reactors. However, studies on neutron irradiation effects of AlN ceramics have been very few. In this study, commercially available AlN ceramics (random orientation) and highly oriented AlN ceramics fabricated under the strong magnetic field were neutron-irradiated, and their defects formation were evaluated from the viewpoint of neutron fluence and irradiation temperature. Furthermore, their recovery behaviors by thermal annealing were also discussed to provide useful experimental data and to obtain systematic and valuable results for fusion applications.

Chapter 1: "Introduction" stated the purpose of this study as well as explained the fundamental interests and importance of ceramics materials for nuclear application. Aluminum nitride (AlN) was introduced in terms of its properties and general applications along with the research interests for the nuclear and fusion reactors fields.

Chapter 2: "Lattice Parameter Changes and Recovery Behavior of Neutron-Irradiated Aluminum Nitride" includes a literature review of neutron irradiation on ceramic's crystal structure and recovery behavior after annealing. Moreover, the initial results on neutron irradiation effects on aluminum nitride (AlN) were introduced. The objectives, specifically in this chapter, were stated. Then, the information on AlN materials, which was used in this study was informed along with the irradiation and annealing processes and operation conditions. In the characterization and analysis section, phase analysis, lattice parameter and lattice strain change, recovery behavior, and isothermal length change by Post-irradiated annealing and activation energy were separately explained in terms of preparation, operation, and analysis method. After that, the result corresponds to each characterization and analysis were reported and discussed, respectively. Finally, the conclusion of the study was explained

The effect on lattice swelling was found differently regarding neutron fluence and irradiation temperature. Irradiated specimens at lower neutron fluence and irradiation temperature responded isotropic unit-cell expansion with greater swelling at a higher dose. On the other hand, irradiated specimens at above  $2 \times 10^{24} \text{ n/m}^2$  ( $E > 0.1 \text{ MeV}$ ), expanded anisotropically as lower in the a-axis and significantly larger in the c-axis which indicated the (0001)-basal plane dislocation. The recovery of the first specimens group started at as same as irradiated temperature, at 523 K and gradually reached the maximum at 1573 K. For the low (up to 573K) and intermediate (573 to 1023 K), the result explained to be involved with the migration of  $V_N$  and  $V_{Al}$ , respectively. Before the saturation of length recovery, the high region (1073 to 1423 K) with high activation energy represents final constant recovery, which supposed that the more complex clustering has occurred in the system. On the other hand, up to 723 K thermal annealing, the anisotropic expanded specimen did not recover indicated almost any dependent interstitials that can be mobile. The same mechanism with the high region of the first group could be explained at 773 to 1123 K as similar energy required was found. At 1173 to 1423 K, rapidly quick and large shrinkage was observed with very high activation energy that might be attributed to forming larger dislocation loops.

Chapter 3: “Highly Oriented Structure Effects of Neutron-Irradiated Aluminum Nitride in Lattice Parameter Changes and Recovery Behavior” introduced the preparation of the material by applying a magnetic field in order to obtain highly oriented structure, and discussed the effect of the crystal structure of neutron-irradiated AlN on their lattice parameter changes and recovery behavior. Firstly, the literature on this technique was reviewed. The objectives, specifically in this chapter, were stated. Then, the information of highly oriented preparation, which was used in this study was informed along with the irradiation and annealing processes and operation conditions. In the characterization and analysis section, phase analysis, degree of orientation, lattice parameter and lattice strain change, and recovery behavior and isothermal length change by Post-irradiated annealing were separately explained in terms of preparation, operation, and analysis method. After that, the result corresponds to each characterization and analysis were reported and discussed, respectively. Finally, the conclusion of the study was explained

Point defects and dislocation loops lying on the (001) plane induced by neutron irradiation are focused on bringing a dramatic change and swelling in the material, so the highly oriented grains is expected as one of the routes to obtaining improved properties. The highly oriented specimens were successfully fabricated under a strong magnetic field for 12 T in the National Institute of Materials Science, Japan. Then, the specimens were neutron-irradiated in the BR2 reactor, and similar thermal annealing and characterization with randomly oriented grain specimens were performed. At low neutron fluence, both random and highly oriented specimen showed small anisotropic expansion. The unit-cell volume expansion of the randomly oriented specimen was almost the same as that of the highly oriented one. Still, at higher neutron fluence, the unit-cell volume expansion of randomly oriented specimen was found significantly larger. In case of length recovery after thermal annealing, both specimens were found similarly started near to the irradiation temperature at around 573 K. The recovery profiles of the specimen with randomly oriented grains and the aligned grains measured along a-axis dominant direction resembled each other. However, the profile result measured along the c-axis direction was different. The larger expansion was found the tended to continue up to the maximum observation temperature.

Chapter 4: “Change in the Appearance and Hardness of Random and Highly Oriented Aluminum Nitride by Neutron Irradiation” introduced the color appearance and transparency change along with the hardening of the materials after neutron irradiation. The color appearance and transparency change were first introduced. The objectives, specifically in this section, were stated. Then, the materials irradiation condition, along with the annealing process and preparation for observation, was explained. The results were finally discussed. After that, the next section of hardening was demonstrated. The previous report of hardening after irradiation was explained. The objective of the hardening study was also stated. Then, the characterization method and results were described and discussed. Finally, the conclusion of this chapter was introduced.

The different types of defects also affected the hardening of the specimen. All specimen was hardened after irradiation, where the specimen without dislocation loops has a higher hardening value. The existence of dislocation caused a reduction in hardening and the weakness of the surface that the indentation quickly generated cracks during the test. The color appearance of specimens also affected and changed from the original translucent yellowish to non-translucent black of non-dislocation, and brown of those with dislocation. Thermal annealing caused the recovery in color. This incident is related to the color center mechanism and can be advantaged to sensor applications.

Chapter 5: “Conclusion” described the overall conclusions precisely of the effect of neutron irradiation on lattice parameter changes and recovery behavior of commercial aluminum nitride with random and highly orientation of particles. Some emphasized effects were stated to provide useful information on aluminum nitride for nuclear and fusion applications.

*Keyword: Aluminum Nitride, Neutron irradiation, Lattice parameter, Dislocation loop, Defects recovery, Activation energy, Grain orientation, strong magnetic field, Color center, hardening*