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研究課題名（和文）Development of a novel multi-directional thermomechanical process to optimize the microstructure of Oxide-Dispersion-Strengthened (ODS) ferritic steel

研究課題名（英文）Development of a novel multi-directional thermomechanical process to optimize the microstructure of Oxide-Dispersion-Strengthened (ODS) ferritic steel

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研究成果の概要（和文）：本研究では、酸化物分散強化鋼の再結晶温度を下げるために、多方向冷間圧延後に熱処理を施すという新しい熱機械的プロセスを開発した。この再結晶工程により、延性およびクリープ特性を向上させることができる。耐照射性を調べるために、イオン照射実験を行った。その結果、冷間圧延により大量の欠陥（転位、粒界など）を誘発することで、照射硬化を抑制できることがわかった。また、室温および673Kでの再結晶試料の照射硬化は照射欠陥の形成によるものであるが、973Kでの照射硬化は確認されなかった。

研究成果の学術的意義や社会的意義
A novel recrystallization process was developed to give rise to a lower recrystallization temperature, homogeneous microstructure, fewer pore defects in matrix, and thus improvements of ductility, creep properties, and formability, which are critical for the fabrication and application of ODS steel.

研究成果の概要（英文）：In this study, a novel thermomechanical process, namely, multi-directional cold rolling followed by thermal annealing, was developed to reduce the recrystallization temperature of oxide-dispersion-strengthened (ODS) steel. This recrystallization process can improve the ductility and creep properties. To investigate the irradiation resistance, ion irradiation experiments were carried out. Results showed that cold rolling can suppress the irradiation hardening by inducing large numbers of defects (dislocations, grain boundaries, etc.). Irradiation hardening of recrystallized samples at room temperature and 673 K was due to the formation of irradiation defects, however, hardening at 973 K was not obviously observed.

研究分野：Fusion Reactor Materials Science

キーワード：ODS steel　Cold rolling　Thermal annealing　Recrystallization　Mechanical properties　Ion irradiation　Irradiation hardening

１．研究開始当初の背景

Oxide-Dispersion-Strengthened (ODS) steel is considered as one of the promising structural materials for fusion reactors, owing to proper high-temperature strength and good irradiation damage resistance. Nano-scale oxide particles are introduced by mechanical alloying and consolidated by hot extrusion. The final products are generally shaped by forging and rolling with subsequent heat treatments. The elongated fine grains with high density of dislocations, and preferential crystal orientation give rise to anisotropic mechanical properties, low ductility, and poor workability, which are recognized as important issues in manufacturing technology for scale-up of the ODS steel. To obtain uniform microstructure, suppress the strength anisotropy, and improve the formability for the following process, recrystallization process is considered. However, recrystallization temperature of as-fabricated 12Cr ODS steel is up to 1673 K, which leads to coarsening of oxide particles and micrometer scale pores formation that significantly degrade the strength. Therefore, a novel thermomechanical process including multi-directional cold rolling and subsequent annealing was developed to alter the original crystal orientation and reduce the recrystallization temperature, which gives fewer pore defects in the matrix. The microstructure was preliminarily evaluated after recrystallization, and further investigation on the mechanical properties and irradiation tolerance is necessary.

２．研究の目的

The purpose of this study is to investigate the high-temperature mechanical properties and the irradiation response of the newly developed ODS steel after the novel recrystallization process.

３．研究の方法

(1) Cold rolling was performed on the transverse section with 90% thickness reduction, and then annealed at 1273 K and 1373 K for 3 h.
(2) Tensile tests were conducted from room temperature to 973 K.
(3) Ion irradiation experiments were carried out at High Fluence Irradiation Facility (HIT), the University of Tokyo, and Dual-Beam Facility for Energy Science and Technology (DuET), Kyoto University. Nano-indentation hardness were measured after irradiation. Irradiated samples were prepared by focused ion beam (FIB) device. Microstructure was characterized by transmission electron microscopy (TEM).

４．研究成果

(1) After the multi-directional cold rolling process, recrystallization occurred at 1273 K and 1373 K, which was lower than the as-fabricated condition and indicated that the multi-directional cold rolling facilitates occurrence of recrystallization at a lower temperature. Moreover, the size and number density of oxide particles were not significantly changed in comparison with the as-fabricated condition.
Fig. 1 shows the tensile properties such as ultimate tensile stress (UTS), yield stress

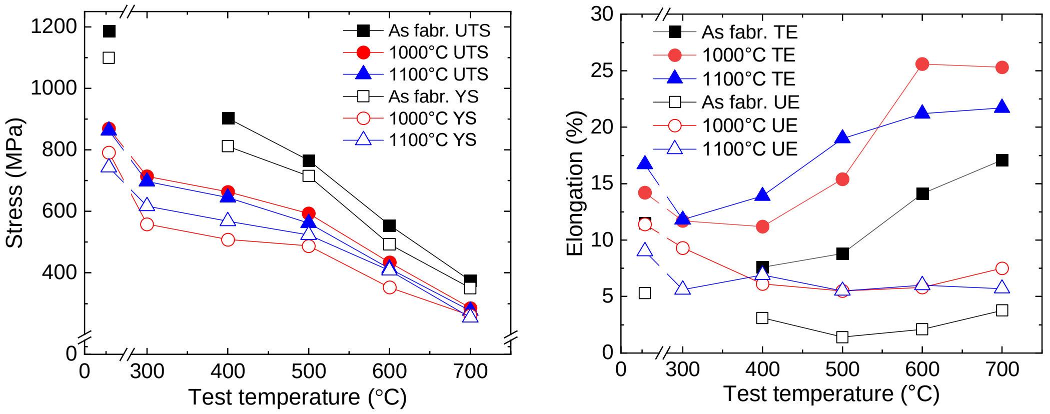

Fig. 1 Tensile properties as a function of temperature of as-fabricated and recrystallized (1273 K and 1373 K) 12Cr ODS steel

(YS), total elongation (TE), and uniform elongation (UE) of as-fabricated (as-fabr) and recrystallized (Rex) 12Cr ODS steel at various temperatures. It showed a similar trend upon temperature that gradually decreased with increasing testing temperature. Tensile strength of recrystallized specimens was lower than the as-fabricated samples, whereas elongation was improved via recrystallization process.
Fig. 2 shows the fracture surfaces of as-fabricated and recrystallized (at 1373 K) 12Cr ODS steel after tensile tests at 973 K. As can be seen, obvious slip and necking behaviors were observed in recrystallized samples after tensile tests.

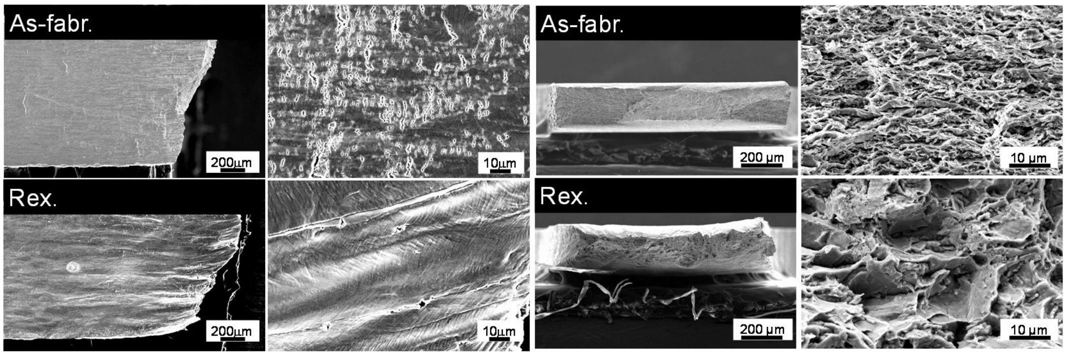

Fig. 2 Fracture surfaces of as-fabricated (as-fabr) and recrystallized (Rex) 12Cr ODS steel after tensile tests at 973 K.

(2) Cold rolled samples with 90% thickness reduction (CR90) and recrystallized (Rex) samples at 1373 K were irradiated by 2.8 MeV Fe^{2+} at room temperature (RT) and 673 K with irradiation dose up to 15 dpa at the peak damage. Fig. 3 shows the nanoindentation hardness of CR90 and Rex at the depth of 150 nm before and after irradiation. Note that hardening was not evidently observed for cold rolled samples, indicating that large plastic deformation inducing high density of dislocations and fine grains can suppress the irradiation hardening. As for the recrystallized samples, the diameter of oxide particles was slightly decreased with increasing the nominal irradiation damage dose, which is probably attributed to the ballistic collisions from irradiation damage cascades. Irradiation hardening of recrystallized specimens was characterized and agreed with the estimation from the contribution of dislocation loops, which suggested that irradiation hardening was caused by the formation of dislocation loops.

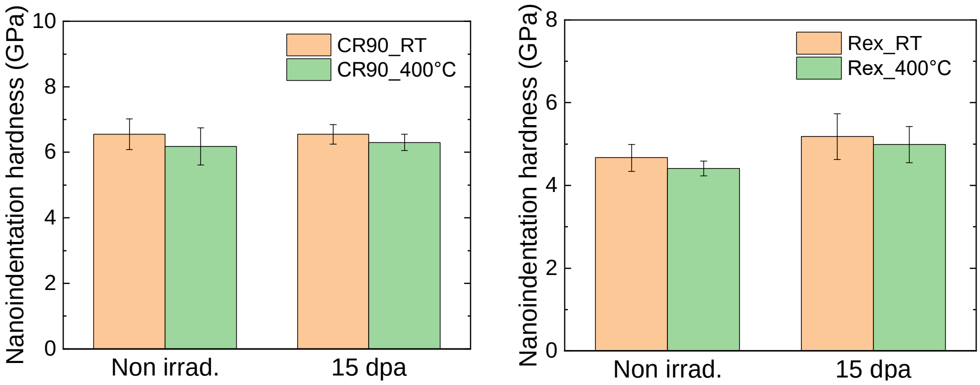

Fig. 3 Nanoindentation hardness of cold rolled (CR90) and recrystallized (Rex) 12Cr ODS steel after ion irradiation at room temperature (RT) and 673 K.

Regarding the dual beam (6.4 MeV Fe^{3+} and 1 MeV He^{+}) irradiation at 973 K up to 10.5 dpa at the peak damage. It showed that irradiation defects such as helium bubbles were observed, however, the irradiation hardening was not obvious, suggesting helium bubbles did not evidently induce hardening at this condition.

５．主な発表論文等

〔雑誌論文〕　計4件（うち査読付論文　4件／うち国際共著　1件／うちオープンアクセス　3件）

１．著者名 Shen Jingjie、Nagasaka Takuya、Muroga Takeo、Yang Huilong、Kano Sho、Abe Hiroaki	４．巻 25
２．論文標題 Microstructural evolution of two-way cold rolled 12Cr ODS steel under 1073 - 1373 K annealing	５．発行年 2020年
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掲載論文のDOI（デジタルオブジェクト識別子） 10.1016/j.nme.2020.100792	査読の有無 有
オープンアクセス オープンアクセスとしている（また、その予定である）	国際共著 －

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掲載論文のDOI（デジタルオブジェクト識別子） 10.1016/j.jnucmat.2020.152606	査読の有無 有
オープンアクセス オープンアクセスではない、又はオープンアクセスが困難	国際共著 －

１．著者名 Shen Jingjie、Nagasaka Takuya、Tokitani Masayuki、Muroga Takeo、Kasada Ryuta、Sakurai Seiji	４．巻 224
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２．論文標題 Microstructure and Mechanical Properties of Y4Zr3012-Added Fe-13.5Cr-2W Oxide-Dispersion-Strengthened Steels, Containing High Contents of C and N, Prepared by Mechanical Alloying and Two-Step Spark Plasma Sintering	５．発行年 2023年
３．雑誌名 Materials	６．最初と最後の頁 2433～2433
掲載論文のDOI（デジタルオブジェクト識別子） 10.3390/ma16062433	査読の有無 有
オープンアクセス オープンアクセスとしている（また、その予定である）	国際共著 該当する

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4．発表年 2023年

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2．発表標題 Ion irradiation response of cold rolled and recrystallized 12Cr ODS steel
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〔図書〕　計0件

〔産業財産権〕

〔その他〕

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6．研究組織

	氏名 （ローマ字氏名） （研究者番号）	所属研究機関・部局・職 （機関番号）	備考

7．科研費を使用して開催した国際研究集会

〔国際研究集会〕　計0件

8．本研究に関連して実施した国際共同研究の実施状況

共同研究相手国	相手方研究機関