



JAEA-Technology

2006-047



JP0650673

Fabrication and Characterization of ^6Li -enriched Li_2TiO_3 Pebbles for a High Li-burnup Irradiation Test

Kunihiko TSUCHIYA and Hiroshi KAWAMURA

Blanket Irradiation and Analysis Group
Fusion Research and Development Directorate

October 2006

Japan Atomic Energy Agency

日本原子力研究開発機構

JAEA-Technology

本レポートは日本原子力研究開発機構が不定期に発行する成果報告書です。

本レポートの入手並びに著作権利用に関するお問い合わせは、下記あてにお問い合わせ下さい。

なお、本レポートの全文は日本原子力研究開発機構ホームページ (<http://www.jaea.go.jp/index.shtml>)
より発信されています。このほか財団法人原子力弘済会資料センター*では実費による複写頒布を行っ
ております。

〒319-1195 茨城県那珂郡東海村白方白根 2 番地 4

日本原子力研究開発機構 研究技術情報部 研究技術情報課

電話 029-282-6387, Fax 029-282-5920

*〒319-1195 茨城県那珂郡東海村白方白根 2 番地 4 日本原子力研究開発機構内

This report is issued irregularly by Japan Atomic Energy Agency

Inquiries about availability and/or copyright of this report should be addressed to

Intellectual Resources Section, Intellectual Resources Department,

Japan Atomic Energy Agency

2-4 Shirakata Shirane, Tokai-mura, Naka-gun, Ibaraki-ken 319-1195 Japan

Tel +81-29-282-6387, Fax +81-29-282-5901

© Japan Atomic Energy Agency, 2006

Fabrication and Characterization of ${}^6\text{Li}$ -enriched Li_2TiO_3 Pebbles for a High Li-burnup Irradiation Test

Kunihiko TSUCHIYA and Hiroshi KAWAMURA⁺¹

Division of Fusion Energy Technology
Fusion Research and Development Directorate
Japan Atomic Energy Agency
Naka-shi, Ibaraki-ken

(Received August 30, 2006)

Lithium titanate (Li_2TiO_3) pebbles are considered to be a candidate material of tritium breeders for fusion reactor from viewpoints of easy tritium release at low temperatures (about 300°C) and chemical stability. In the present study, trial fabrication tests of ${}^6\text{Li}$ -enriched Li_2TiO_3 pebbles of 1mm in diameter were carried out by a wet process with a dehydration reaction, and characteristics of the ${}^6\text{Li}$ -enriched Li_2TiO_3 pebbles were evaluated for preparation of a high Li-burnup test in a testing reactor. Powder of 96at% ${}^6\text{Li}$ -enriched Li_2TiO_3 was prepared by a solid state reaction, and two kinds of ${}^6\text{Li}$ -enriched Li_2TiO_3 pebbles, namely un-doped and TiO_2 -doped Li_2TiO_3 pebbles, were fabricated by the wet process. Based on results of the pebble fabrication tests, two kinds of ${}^6\text{Li}$ -enriched Li_2TiO_3 pebbles were successfully fabricated with target values (density : 80-85%T.D., grain size : $<5\mu\text{m}$, diameter : 0.85-1.18mm). Physical, chemical and mechanical properties of these pebbles were also evaluated before neutron irradiation tests. Sphericity of these Li_2TiO_3 pebbles was a satisfying value of about 1.05. Contact strength of these pebbles was about 6300MPa, which was almost the same as that of the Li_2TiO_3 pebbles with natural Li.

Keywords: Lithium Titanate (Li_2TiO_3), Fusion Reactor, Breeding Blanket, Tritium Breeder, Wet Process, Irradiation Test, Characterization

⁺¹: Policy Planning and Administration Department

高 Li 燃焼照射試験のための ${}^6\text{Li}$ 濃縮 Li_2TiO_3 微小球の製造と特性評価

日本原子力研究開発機構核融合研究開発部門

核融合エネルギー工学研究開発ユニット

土谷 邦彦・河村 弘⁺¹

(2006 年 8 月 30 日受理)

チタン酸リチウム (Li_2TiO_3) 微小球は、低い温度 (約 300°C) における容易なトリチウム放出及び化学的安定性の観点から核融合炉のためのトリチウム増殖材料の候補材料として考えられている。本研究では、直径 1mm の ${}^6\text{Li}$ 濃縮 Li_2TiO_3 微小球の試作試験を脱水反応を利用した湿式法 (脱水型ゲル化法) により行い、試験炉を用いた高 Li 燃焼試験の準備のためにその ${}^6\text{Li}$ 濃縮 Li_2TiO_3 微小球の特性を評価した。96at%の ${}^6\text{Li}$ 濃縮度を有する Li_2TiO_3 粉末を固相反応により準備し、 TiO_2 の無添加のものと TiO_2 を添加したものの 2 種類の Li_2TiO_3 微小球をこの湿式法により製作した。微小球製造試験の結果に基づき、目標値 (焼結密度: 80-85%T.D., 結晶粒径: $5\mu\text{m}$ 以下, 直径: $0.85\sim 1.18\text{mm}$) を有する 2 種類の ${}^6\text{Li}$ 濃縮 Li_2TiO_3 微小球を製造することに成功した。また、これら 2 種類の物理的、化学的及び機械的特性を中性子照射試験前に評価した。これらの Li_2TiO_3 微小球の真球度は約 1.05 であった。また、微小球の接触強度は約 6300MPa であり、天然同位体比を有する Li を用いた Li_2TiO_3 微小球の強度とほぼ同じであった。

CONTENTS

1. Introduction	1
2. Experimental	1
2.1 Preparation of ^6Li -enriched Li_2TiO_3 powder	1
2.2 Fabrication of ^6Li -enriched Li_2TiO_3 pebbles	2
3. Results and Discussions	2
3.1 Preparation of ^6Li -enriched Li_2TiO_3 powder	2
3.2 Fabrication tests of ^6Li -enriched Li_2TiO_3 pebbles	2
3.3 Characterization of ^6Li -enriched Li_2TiO_3 pebbles	3
4. Conclusions	4
Acknowledgments	4
References	4
Appendix A Preparation of ^6Li -enriched Li_2TiO_3 Pellets	13

目 次

1. 序 論	1
2. 実 験	1
2.1 ^6Li 濃縮 Li_2TiO_3 粉末の調整	1
2.2 ^6Li 濃縮 Li_2TiO_3 微小球の製造	2
3. 結果と議論	2
3.1 ^6Li 濃縮 Li_2TiO_3 粉末の調整	2
3.2 ^6Li 濃縮 Li_2TiO_3 微小球の製造試験	2
3.3 ^6Li 濃縮 Li_2TiO_3 微小球の特性評価	3
4. 結 論	4
謝 辞	4
参考文献	4
付録 A ^6Li 濃縮 Li_2TiO_3 ペレットの準備	13

1. Introduction

Recently, lithium titanate (Li_2TiO_3) has attracted attention of many researchers from viewpoints of easy tritium release at low temperatures ($\sim 300^\circ\text{C}$), chemical stability and low tritium inventory [1-3]. A small Li_2TiO_3 pebble is selected in the Japanese [4] and European [5] designs of the fusion blanket. As the fabrication method of Li_2TiO_3 pebbles, the wet process is the most advantageous from viewpoints of mass fabrication and lithium recycling [6-7].

Irradiation damage and tritium breeder ratio (TBR) of Li_2TiO_3 pebbles with a structure of a DEMO breeding blanket were evaluated by the calculation codes (the MCNP code, etc.) [8]. According to the calculation results, Li-burnup of Li_2TiO_3 pebbles was about 20% in DEMO conditions. However, characterization of Li_2TiO_3 pebbles has not been performed in high Li-burnup irradiation tests. Therefore, an irradiation test with post-irradiation examinations (PIEs) of Li_2TiO_3 pebbles and pellets have been carried out in Kazakhstan National Nuclear Center as a project (K-578) of the International Science and Technology Center (ISTC) between 2002 and 2008 [9].

In the present study, trial fabrication tests of ^6Li -enriched Li_2TiO_3 pebbles of 1mm in diameter were carried out by the wet process with a dehydration reaction, and characteristics of these pebbles were evaluated before a high Li-burnup irradiation test.

2. Experimental

2.1. Preparation of ^6Li -enriched Li_2TiO_3 powder

Powder of 96at% ^6Li -enriched Li_2TiO_3 was prepared by a solid state reaction between ^6Li -enriched lithium carbonate (Li_2CO_3) and titanium oxide (TiO_2) powders. The reaction for preparation of the Li_2TiO_3 powder is expressed by Eq.(1).



Each powder (Li_2CO_3 and TiO_2) was mixed, and the mixed powder was pulverized in a ball mill using ethanol. After drying, the mixed powder was reacted in air at $700\sim 800^\circ\text{C}$ for 24 h. After the reaction, the powder was pulverized again in ethanol, and was reacted in air at 900°C for 4 h. This ^6Li -enriched Li_2TiO_3 powder was pulverized by a jet mill after the reaction. Chemical compositions of the ^6Li -enriched Li_2CO_3 powder and the TiO_2 powder are shown in Table 1 and Table 2, respectively.

Crystal structure of the ^6Li -enriched Li_2TiO_3 powder was analyzed by X-ray diffractometry (XRD), and particle size was measured by a laser diffraction method. Impurities were analyzed by atomic emission spectrometry with inductively coupled plasma (ICP-AES), atomic absorption spectrometry (AAS) and ICP-Mass Spectrometer (ICP-MS). Enrichment of ^6Li for this powder was also measured by ICP-MS.

2.2 Fabrication of ^6Li -enriched Li_2TiO_3 pebbles

Pebbles of un-doped Li_2TiO_3 and TiO_2 -doped Li_2TiO_3 were fabricated using ^6Li -enriched lithium by the wet process with a dehydration reaction. The TiO_2 powder was added for suppression grain growth during sintering. A flow chart for the fabrication process is shown in Fig.1. This procedure includes a fabrication process of gel-spheres, and subsequent dropping, drying, calcination and sintering processes, as follows.

- 1) Fabrication of gel-spheres: a liquid mixture was prepared from ^6Li -enriched Li_2TiO_3 powder, TiO_2 powder, polyvinyl-alcohol (PVA) and water. The mixture was dropped through the nozzles into acetone. At this time, the liquid mixture changed by itself to spheres due to its surface tension, and the spheres were gelled in acetone by a dehydration reaction. Thereafter, the gel-spheres were dried in air.
- 2) Calcination of gel-spheres: The dried gel-spheres were calcined at 600°C in air to remove PVA, and low-density Li_2TiO_3 pebbles were fabricated.
- 3) Sintering: The low-density Li_2TiO_3 pebbles were sintered at $1000\text{--}1300^\circ\text{C}$ in air to increase their density.

Characteristics of the un-doped Li_2TiO_3 pebbles and the TiO_2 -doped Li_2TiO_3 pebbles were evaluated. Density of the Li_2TiO_3 pebbles was measured by mercury porosimetry. Microstructure was observed by scanning electron microscopy (SEM) and a photographic analysis equipment. The crystal structure was analyzed by XRD. Impurities in the Li_2TiO_3 pebbles were measured by ICP-AES, AAS and ICP-MS. Enrichment of ^6Li for the Li_2TiO_3 pebbles was also measured by ICP-MS. Collapse loads of the Li_2TiO_3 pebbles were measured with an unconfined compression tester having a compression indenter made of SiC.

3. Results and Discussions

3.1 Preparation of ^6Li -enriched Li_2TiO_3 powder

Surface appearance and particle size distribution of the ^6Li -enriched Li_2TiO_3 powder are shown in Fig.2 and Fig.3, respectively. The average particle size of this powder was about $0.72\text{ }\mu\text{m}$. An XRD pattern of the ^6Li -enriched Li_2TiO_3 powder is shown in Fig.4. Detected X-ray diffraction peaks of the powder were corresponded with the diffraction peaks of Li_2TiO_3 , and no diffraction peaks of Li_2CO_3 and TiO_2 were detected. Chemical composition of the ^6Li -enriched Li_2TiO_3 powder is shown in Table 3.

3.2 Fabrication tests of ^6Li -enriched Li_2TiO_3 pebbles

In the first examination, the relationship between the content of Li_2TiO_3 powder in the mixture liquid and the diameter of the fabricated pebbles was studied, where Li_2TiO_3 powder containing Li with natural abundance (7.5at%) of ^6Li (average particle size : $0.62\text{ }\mu\text{m}$) was used [10]. In this test, content of PVA was selected as 5wt%. The result is shown in Fig.5. The pebble diameter increased with increasing the content of Li_2TiO_3 powder. Since the Li_2TiO_3 pebbles of 1mm in diameter have a target diameter range

of 0.85 to 1.18mm, the content of Li_2TiO_3 powder in the liquid mixture was selected as $25 \pm 5\text{wt}\%$, corresponding to this target diameter range. The fabrication of Li_2TiO_3 pebbles with 1mm in diameter was tried with 96at% ^6Li -enriched Li_2TiO_3 powder (average particle size : $0.72\mu\text{m}$). The result is also shown in Fig.5. The diameter of these pebbles (96at% ^6Li -enriched) was 1.00 mm for a selected Li_2TiO_3 content of 25wt%.

In the second examination, the relationship between the sintering temperature and the pebble density was studied. Mixed powder of 96at% ^6Li -enriched Li_2TiO_3 and TiO_2 (purity : 99.99%, particle size : $0.2\text{--}2\mu\text{m}$) was used in this test. Contents of PVA and Li_2TiO_3 powder were 5 and 25%, respectively. The pebble density increased with increasing the sintering temperature. The density of 5mol% TiO_2 -doped Li_2TiO_3 pebbles was larger than that of un-doped Li_2TiO_3 pebbles sintered at the same temperature. Since the un-doped Li_2TiO_3 pebbles and the TiO_2 -doped Li_2TiO_3 pebbles have a target density in the range of 80 to 85%T.D., the sintering temperatures for the target density were selected as 1200°C for the un-doped Li_2TiO_3 pebbles and 1150°C for the 5mol% TiO_2 -doped Li_2TiO_3 pebbles, respectively. Fabrication of Li_2TiO_3 pebbles with 1 mm in diameter was tried with the 96at% ^6Li -enriched Li_2TiO_3 powder, and the result is shown in Fig.6.

3.3 Characterization of ^6Li -enriched Li_2TiO_3 pebbles

Characteristics of the un-doped and the TiO_2 -doped Li_2TiO_3 pebbles with ^6Li -enriched lithium are summarized in Table 4. Results of XRD in Fig.7 indicate that the detected diffraction peaks of the fabricated pebbles correspond to those of Li_2TiO_3 .

Photographs of the Li_2TiO_3 pebbles and the 5mol% TiO_2 -doped Li_2TiO_3 pebbles are shown in Fig.8. Each pebble was sifted through screens of 0.85 and 1.17 mm, and average diameters of the Li_2TiO_3 pebbles and the 5mol% TiO_2 -doped Li_2TiO_3 pebbles were 1.00 and 0.99 mm, respectively. Diameter distributions of ^6Li -enriched Li_2TiO_3 pebbles and 5mol% TiO_2 -doped Li_2TiO_3 pebbles are shown in Fig.9. Sphericity of the Li_2TiO_3 pebbles, which is the ratio of the longest diameter to the shortest diameter, was less than 1.05. This value is satisfactory at this stage. Densities of the Li_2TiO_3 pebbles and the TiO_2 -doped Li_2TiO_3 pebbles were 84.2 and 82.2%T.D., respectively; they were well controlled within the target range by adopting the wet process with a dehydration reaction.

SEM photographs and analytical photographs of these pebbles are shown in Fig.10, and grain sizes of the ^6Li -enriched Li_2TiO_3 pebbles calculated with the analytical photograph were less than $5\mu\text{m}$.

The contact strength was evaluated by Hertzian contact theory [11]. The average strength of the Li_2TiO_3 pebbles and the 5mol% TiO_2 -doped Li_2TiO_3 pebbles were 6.5×10^3 and 6.2×10^3 MPa, respectively. The contact strength of these ^6Li -enriched pebbles was the same as that of Li_2TiO_3 pebbles with natural Li, and was independent of ^6Li enrichment. Finally, impurities of these pebbles are shown in Table 5. In these elements, impurity sodium was detected in large amounts. The cause of this result is thought to be that sodium in PVA remained in the fabricated pebbles.

4. Conclusions

The present study revealed the relationship between the content of Li_2TiO_3 powder in the mixture liquid and the pebble diameter, as well as that between the sintering temperature and the pebble density. The pebble diameter and the pebble density were well controlled in the target ranges by the wet process. Based on the results, the un-doped Li_2TiO_3 pebbles and the TiO_2 -doped Li_2TiO_3 pebbles with the target values (density : 80-85%T.D., grain size $<5\mu\text{m}$, diameter : 0.85-1.18mm) were successfully fabricated by the wet process. The sphericity was satisfying value of less than 1.05.

The ^6Li -enriched Li_2TiO_3 pebbles and the TiO_2 -doped, ^6Li -enriched Li_2TiO_3 pebbles have been irradiated in a material testing reactor, and characterization of these pebbles has been made under neutron irradiation. The results will be described elsewhere.

Acknowledgments

The authors would appreciate specialists in NGK Insulators, LTD. (fabrication of the ^6Li -enriched Li_2TiO_3 powder and ^6Li -enriched Li_2TiO_3 pellets) and Nuclear Fuel Industries, Ltd. (fabrication of the ^6Li -enriched Li_2TiO_3 pebbles) for their cooperation in the achievement of this study.

References

- [1] P. A. Finn, K. Kurasawa, S. Nasu, K. Noda, T. Takahashi, H. Takeshita, T. Tanifuji, H. Watanabe, Proc. 9th IEEE Symp. on Engineering Problems of Fusion Research, Vol.II, (1981) pp.1200-1204.
- [2] P. Gierszewski, Fusion Eng. Des. **39-40** (1998) 739-743.
- [3] T. Kawagoe, M. Nishikawa, A. Baba, S. Beloglazov, J. Nucl. Mater. **297** (2001) 27-34.
- [4] M. Enoda, Y. Ohara, M. Akiba, et al., JAERI-Tech 2001-078 (2001) [in Japanese].
- [5] M. Gasparotto, L.V. Boccaccini, L. Giancarli, S. Malang, Y. Poitevin, Fusion Eng. Des. **61-62** (2002) 263-271.
- [6] K. Tsuchiya, H. Kawamura, K. Fuchinoue, H. Sawada, K. Watarumi, J. Nucl. Mater. **258-263** (1998) 1985-1990.
- [7] K. Tsuchiya, H. Kawamura, J. Nucl. Mater. **283-287** (2000) 1380-1384.
- [8] U. Fischer, S. Herring, A. Hogenbirk, D. Leichtle, Y. Nagao, B.J. Pijlgrims, A. Ying, Proc. 8th International Workshop on Ceramic Breeder Blanket Interactions (CBBI-8), pp.248-280 (1999)
- [9] Y. Chikhray, V. Shestakov, T. Kulsartov, I. Tazhibayeva, H. Kawamura, A. Kuykabaeva, presented at the 12th International Conference on Fusion Reactor Materials (ICFRM-12), Dec. 4-9, 2005, Santa Barbara, California, U.S.A., to be published in J. Nucl. Mater.
- [10] K. Tsuchiya, T. Hoshino, H. Kawamura, T. Takayama, J. Ceram. Soc. Japan, Supplement 112-1, 112 (2004) S183-S186.
- [11] Japan Society of Mechanical Engineers, Materials and Mechanics Handbook, 1999 (in Japanese).

Table 1 Chemical composition of ^6Li -enriched Li_2CO_3 powder.

Purity	99.45%					
Elements	Ca	Na	K	Mg	B	Co
Content (wt%)	<0.01	<0.005	<0.01	<0.0005	<0.002	<0.00005
Elements	Al	Zr	Fe	U		
Content (wt%)	0.0050	<0.0001	0.0005	-		
^6Li enrichment	96at%					

Table 2 Chemical composition of TiO_2 powder.

Purity	99.96%					
Elements	Al_2O_3	Fe_2O_3	SiO_2	P_2O_5	Na_2O	Sb_2O_3
Content (wt%)	0.004	0.0022	0.016	0.000	0.003	0.0000
Elements	V_2O_5	Nb_2O_5	Ni	Cr_2O_3	CaO	As
Content (wt%)	0.0001	0.001	0.0002	0.0005	0.009	0.0001
Elements	Pb	Zn	K_2O	SO_3	Cl	C
Content (wt%)	0.0000	0.0001	0.000	0.00	0.002	0.05

Table 3 Chemical composition and ^6Li -enrichment of Li_2TiO_3 powder.

Elements	Ca	Na	K	Mg	B	Co
Content (wt%)	<0.01	0.011	<0.0001	<0.0001	<0.01	0.0021
Elements	Al	Zr	Fe	U		
Content (wt%)	0.0050	<0.0001	0.0061	<0.0001		
^6Li enrichment	96at%					

Table 4 Characteristics of ^6Li -enriched Li_2TiO_3 and TiO_2 -doped ^6Li -enriched Li_2TiO_3 pebbles.

Properties	Measured values		Measuring method
	0mol% TiO_2 ^6Li -enriched Li_2TiO_2	5mol% TiO_2 ^6Li -enriched Li_2TiO_2	
Density	84.2 %T.D.	82.2%T.D.	Liquid Immersion Method (Hg)
^6Li enrichment	96 at%	96 at%	ICP-MS Analysis
Pebble diameter	$\phi 0.85 - 1.18$ mm ($\phi 1.00$ mm av.)	$\phi 0.85 - 1.18$ mm ($\phi 0.99$ mm av.)	Sieve Classification
Sphericity	1.05	1.05	Photograph
Grain size	4.0 μm av.	2.8 μm av.	SEM observation
Contact strength	6.5×10^3 MPa	6.2×10^3 MPa	Collapsed load test

Table 5. Impurities in ^6Li -enriched Li_2TiO_3 pebbles and TiO_2 -doped ^6Li -enriched Li_2TiO_3 pebbles.

Elements	Contents (wt%)	
	Un-doped ^6Li -enriched Li_2TiO_2	5mol% TiO_2 ^6Li -enriched Li_2TiO_2
Li	11.5	10.9
Ti	44.5	44.0
Ca	< 0.01	< 0.01
Na	0.014	0.017
K	< 0.0001	< 0.0001
Mg	0.0005	0.0004
B	< 0.01	< 0.01
Co	0.0032	0.0028
Al	0.0085	0.0058
Zr	0.0003	0.0002
Fe	0.0066	0.0055
U	< 0.0001	< 0.0001

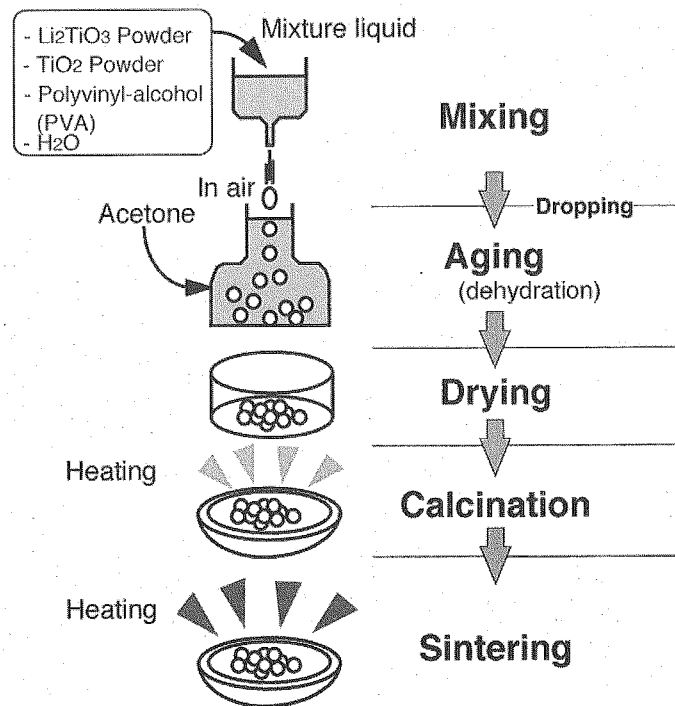


Fig. 1 Flow chart of fabrication of Li_2TiO_3 pebbles by the wet process with a dehydration reaction.

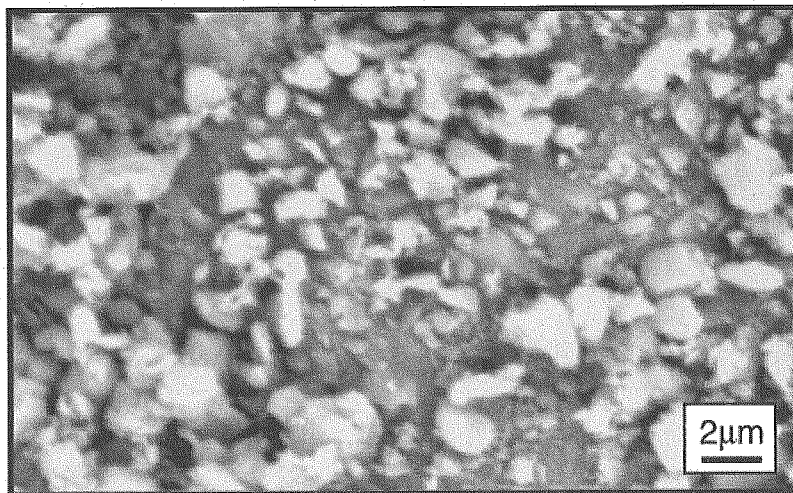


Fig.2 Photograph of ^6Li -enriched Li_2TiO_3 powder.

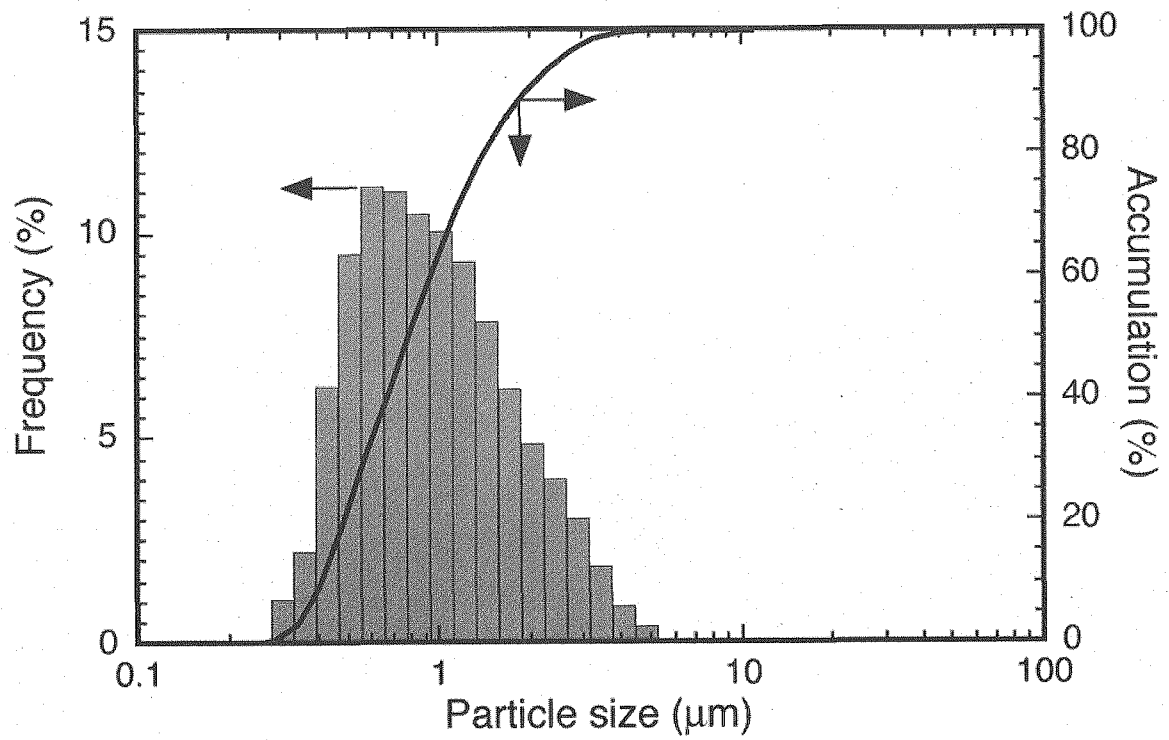


Fig.3 Distribution of particle size of ${}^6\text{Li}$ -enriched Li_2TiO_3 powder.

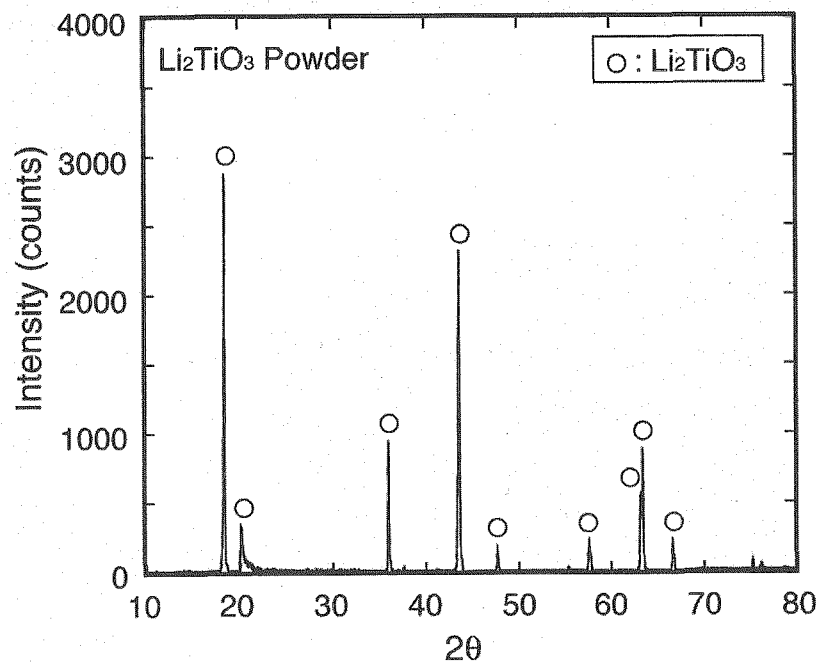


Fig.4 XRD pattern of ${}^6\text{Li}$ -enriched Li_2TiO_3 powder.

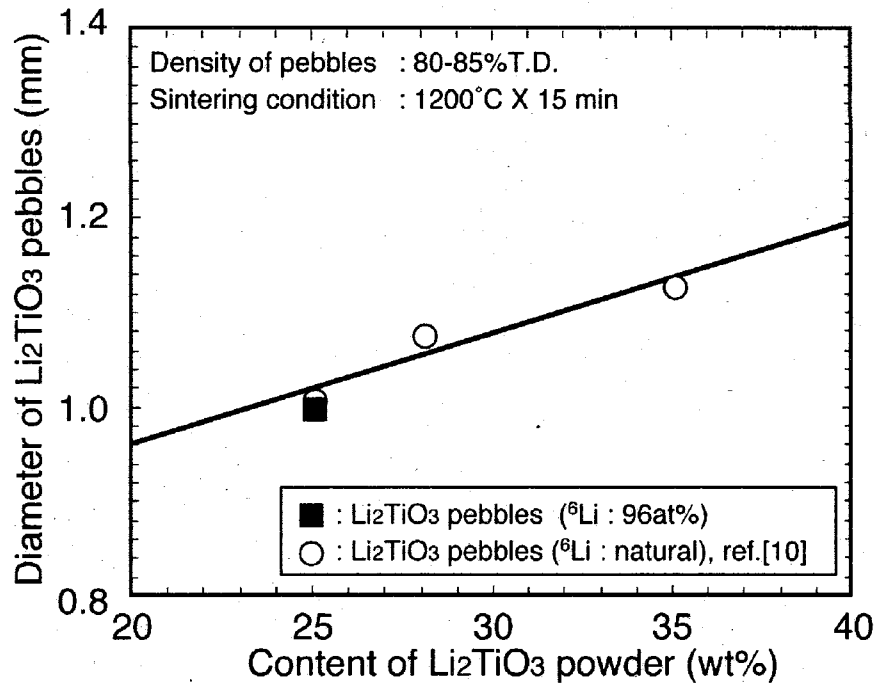


Fig.5 Relationship between content of Li_2TiO_3 powder in mixture liquid and pebble diameter.

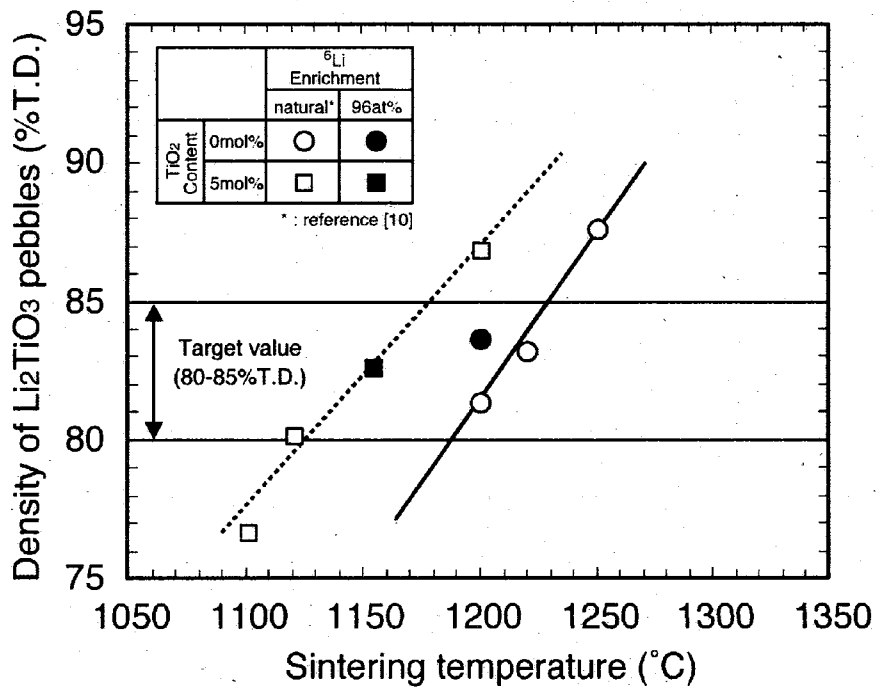


Fig.6 Relationship between sintering temperature and pebble density.

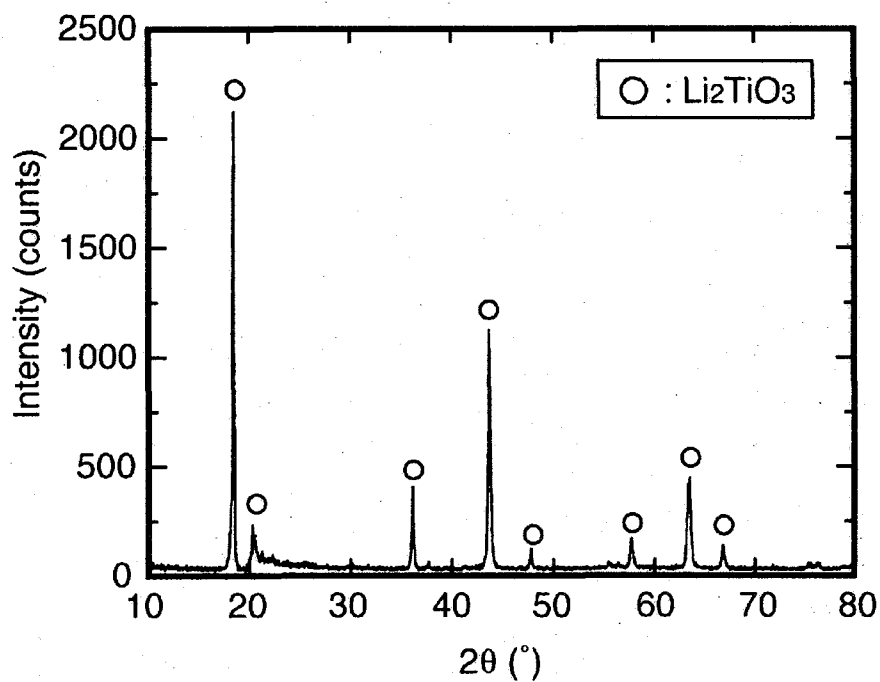
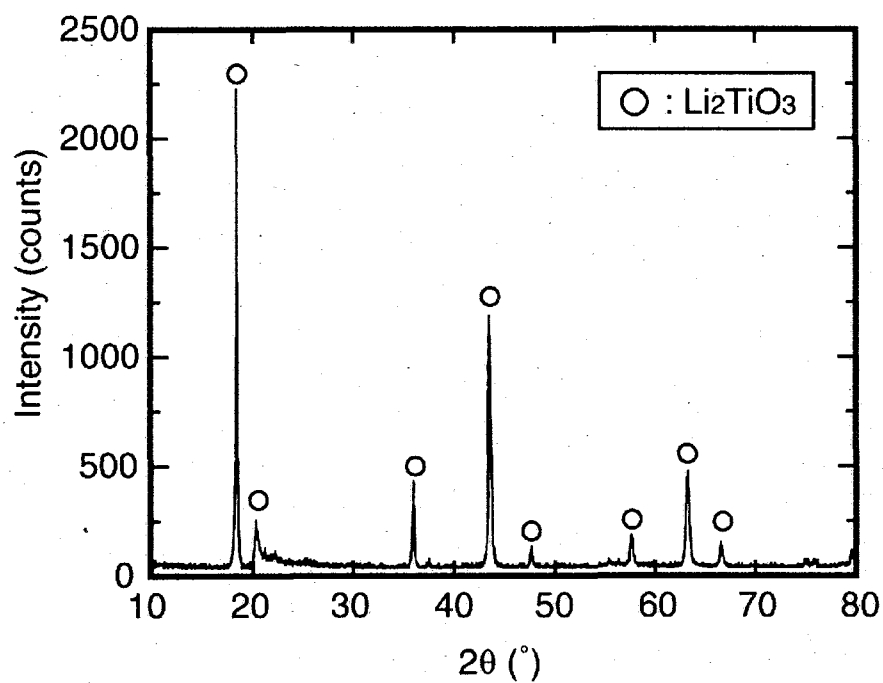
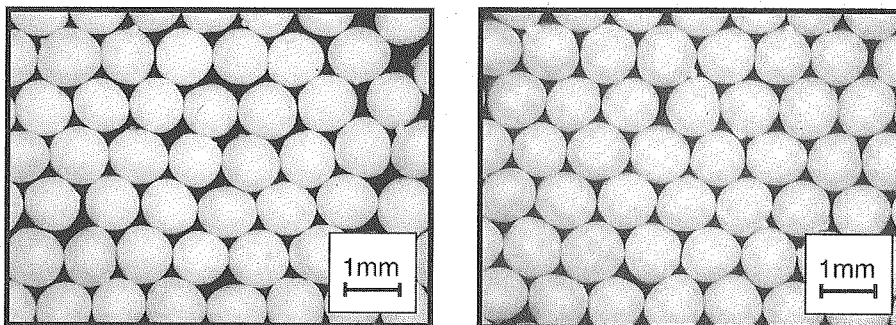
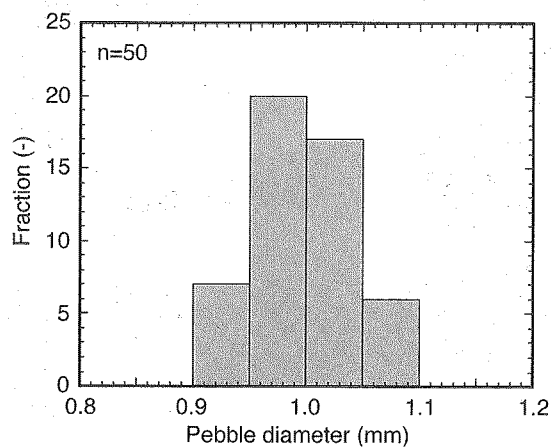
(a) ^6Li -enriched Li_2TiO_3 pebbles (un-doped)(b) TiO_2 -doped ^6Li -enriched Li_2TiO_3 pebbles

Fig.7. X-ray diffraction patterns of ^6Li -enriched Li_2TiO_3 pebbles and TiO_2 -doped ^6Li -enriched Li_2TiO_3 pebbles.

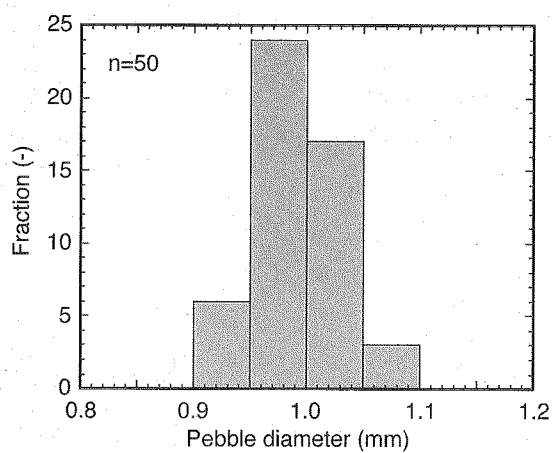


(1) Li_2TiO_3 pebbles (un-doped) (2) 5mol% TiO_2 -doped Li_2TiO_3 pebbles

Fig. 8 Photographs of ^6Li -enriched Li_2TiO_3 pebbles.



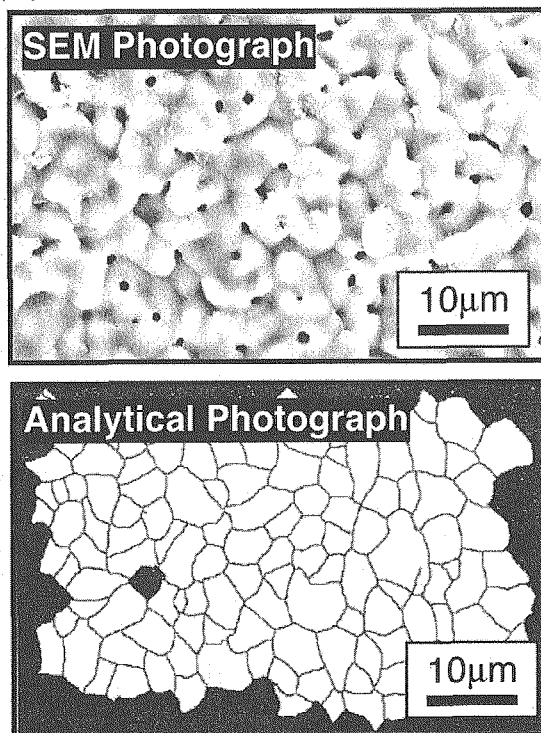
(a) ^6Li -enriched Li_2TiO_3 pebbles (un-doped)



(b) TiO_2 -doped ^6Li -enriched Li_2TiO_3 pebbles

Fig.9 Distributions of pebble diameter of ^6Li -enriched Li_2TiO_3 pebbles.

(a) Li_2TiO_3 pebbles



(b) TiO_2 -doped Li_2TiO_3 pebbles

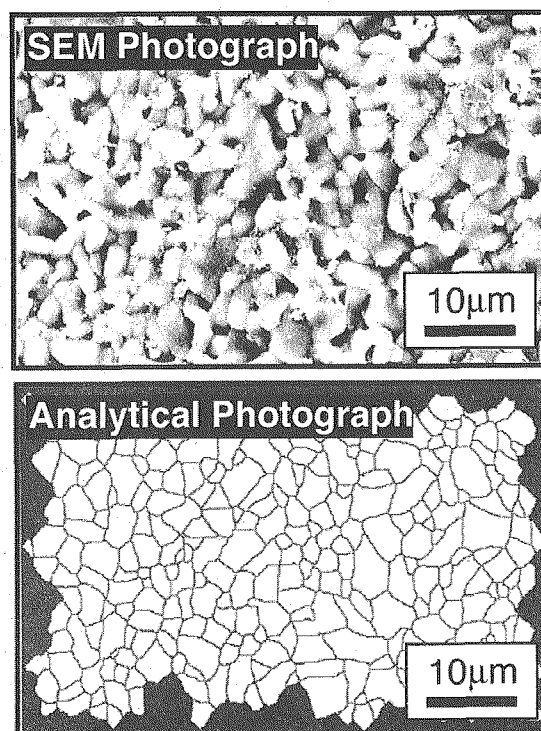


Fig.10 SEM and analytical photographs of ^6Li -enriched Li_2TiO_3 pebbles.

Appendix A: Preparation of ^6Li -enriched Li_2TiO_3 pellets

A-1. Introduction

An irradiation test with post-irradiation examinations of Li_2TiO_3 pellets has been carried out in Kazakhstan National Nuclear Center as a regular project (K-578) of the International Science and Technology Center (ISTC) between 2002 and 2008. This appendix describes preparation of ^6Li -enriched Li_2TiO_3 pellets carried out by a cold pressing/sintering process, and characteristics of these pellets evaluated before the high Li-burnup irradiation test.

A-2. Experimental

Pellets of Li_2TiO_3 and TiO_2 -doped Li_2TiO_3 were fabricated for measurement of swelling, tritium release and thermal properties.

Powder of ^6Li -enriched Li_2TiO_3 was prepared by a solid state reaction between ^6Li -enriched lithium carbonate (Li_2CO_3) and titanium oxide (TiO_2) powders. Characteristics of the ^6Li -enriched Li_2TiO_3 powder were the same as those of the powder for pebble fabrication. The preparation flow chart of the Li_2TiO_3 pellets is shown in Fig.A-1, and main steps are as follows:

- 1) Preparation of powders: mixing of the starting materials (Li_2TiO_3 and TiO_2 powders), drying and sieving.
- 2) Fabrication of pellets: cold pressing and sintering.

Un-doped Li_2TiO_3 , 5mol% TiO_2 -doped Li_2TiO_3 and 10mol% TiO_2 -doped Li_2TiO_3 pellets were fabricated. The dimension of these prepared pellets was 8 mm in diameter and 2 mm in thickness. Density of the un-doped Li_2TiO_3 and the 10mol% TiO_2 -doped Li_2TiO_3 pellets was 83 ± 2 %T.D. Densities of the 5mol% TiO_2 -doped Li_2TiO_3 pellets were 78 ± 2 , 83 ± 2 and 88 ± 2 %T.D. for different production batches. The crystal structure of the ^6Li -enriched Li_2TiO_3 pellets was analyzed by XRD and particle size was measured by SEM observation. Impurities were measured by ICP-AES, AAS and ICP-MS. ^6Li enrichment of these pellets was also measured by ICP-MS.

A-3. Results

The relationship between the sintering temperature and the density of the pellets was studied with the 96at% ^6Li -enriched Li_2TiO_3 powder and the TiO_2 powder (purity : 99.99%, particle size : 0.2-2 μm). The result of this test is shown in Fig.A-2. Contents of the binder were 0 and 2.5%. The sintered density of the pellets increased with increasing the sintering temperature. The density of the TiO_2 -doped Li_2TiO_3 pellets was larger than that of un-doped Li_2TiO_3 pellets sintered at the same temperature.

Characteristics of ^6Li -enriched Li_2TiO_3 pebbles and TiO_2 -doped ^6Li -enriched Li_2TiO_3 pebbles are summarized in Table A-1. Results of XRD in Fig.A-3 indicate that the detected diffraction peaks of the fabricated pebbles correspond to those of Li_2TiO_3 .

Photographs of Li_2TiO_3 , 5mol% TiO_2 -doped Li_2TiO_3 and 10mol% TiO_2 -doped Li_2TiO_3 pellets are shown in Fig.A-4. A SEM photograph of these pellets are shown in Fig.A-5, and grain sizes of ^6Li

enriched Li_2TiO_3 pellets were less than $2\mu\text{m}$.

A-4. Conclusions

Based on these fabrication tests, the ^6Li -enriched Li_2TiO_3 and the TiO_2 -doped ^6Li -enriched Li_2TiO_3 pellets with the target values (density : 80-85%T.D., grain size $<5\mu\text{m}$) were successfully fabricated by the cold pressing/sintering process.

The ^6Li -enriched Li_2TiO_3 and the TiO_2 -doped ^6Li -enriched Li_2TiO_3 pellets have been irradiated in a material testing reactor, and characterization of these pellets has been made under neutron irradiation.

Table A-1 Characteristics of ^6Li -enriched Li_2TiO_3 and TiO_2 -doped ^6Li -enriched Li_2TiO_3 pellets.

Properties	Measured values	Measuring method
Density	78 - 88 %T.D.	Calculation from measured values by size and weight
^6Li enrichment	96at%	ICP-MS
TiO_2 doping	0, 5 and 10 mol%	
Dimension	$\phi 8 \times 2$ mm	Measurement by micrometer
Crystal structure	(see Fig.A-2)	XRD Analysis
Grain size	$\sim 1.5 \mu\text{m}$	SEM observation
Chemical composition	(see Table A-2)	ICP-AES, ICP-MS, AAS

Table A-2. Impurities in ^6Li -enriched Li_2TiO_3 and TiO_2 -doped ^6Li -enriched Li_2TiO_3 pellets.

(Unit: wt%)

Elements	Un-doped Li_2TiO_3 83%T.D.	5mol% TiO_2 -doped Li_2TiO_3 78%T.D.	5mol% TiO_2 -doped Li_2TiO_3 83%T.D.	5mol% TiO_2 -doped Li_2TiO_3 88%T.D.	10mol% TiO_2 -doped Li_2TiO_3 83%T.D.
Li	11.6	11.1	11.2	11.2	10.7
Ti	43.6	44.2	44.4	44.4	44.9
Ca	<0.01	<0.01	<0.01	<0.01	<0.01
B	<0.01	<0.01	<0.01	<0.01	<0.01
Zr	0.0006	0.0009	0.0009	0.0009	0.0012
U	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Na	0.010	0.014	0.013	0.0054	0.0078
K	0.0005	0.0015	0.0015	0.0007	0.0024
Mg	0.0007	0.0008	0.0009	0.0004	0.0007
Co	0.0027	0.0020	0.0025	0.0023	0.0025
Al	0.0070	0.0054	0.0066	0.0065	0.014
Fe	0.0089	0.0073	0.0070	0.0051	0.0088

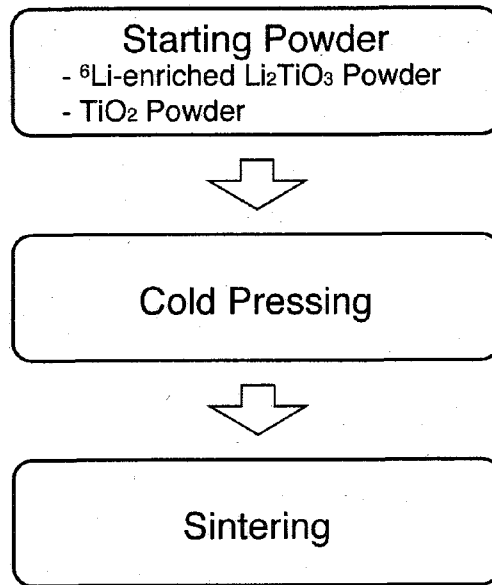


Fig. A-1 Flow chart of fabrication of Li_2TiO_3 pellets.

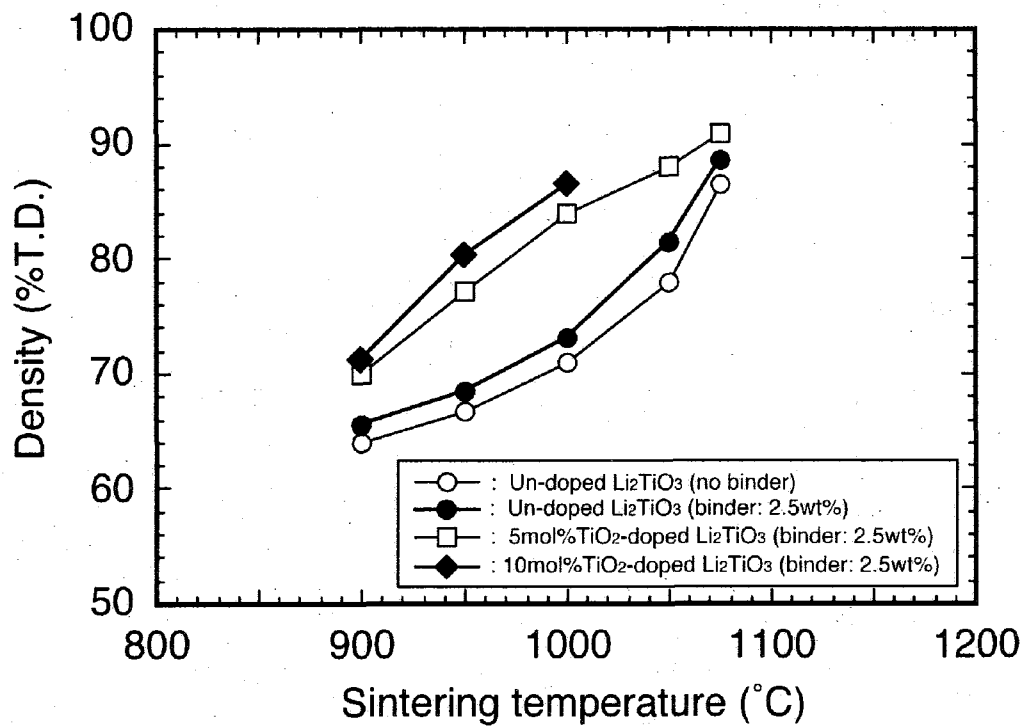


Fig.A-2 Relationship between sintering temperature and density of Li_2TiO_3 pellets.

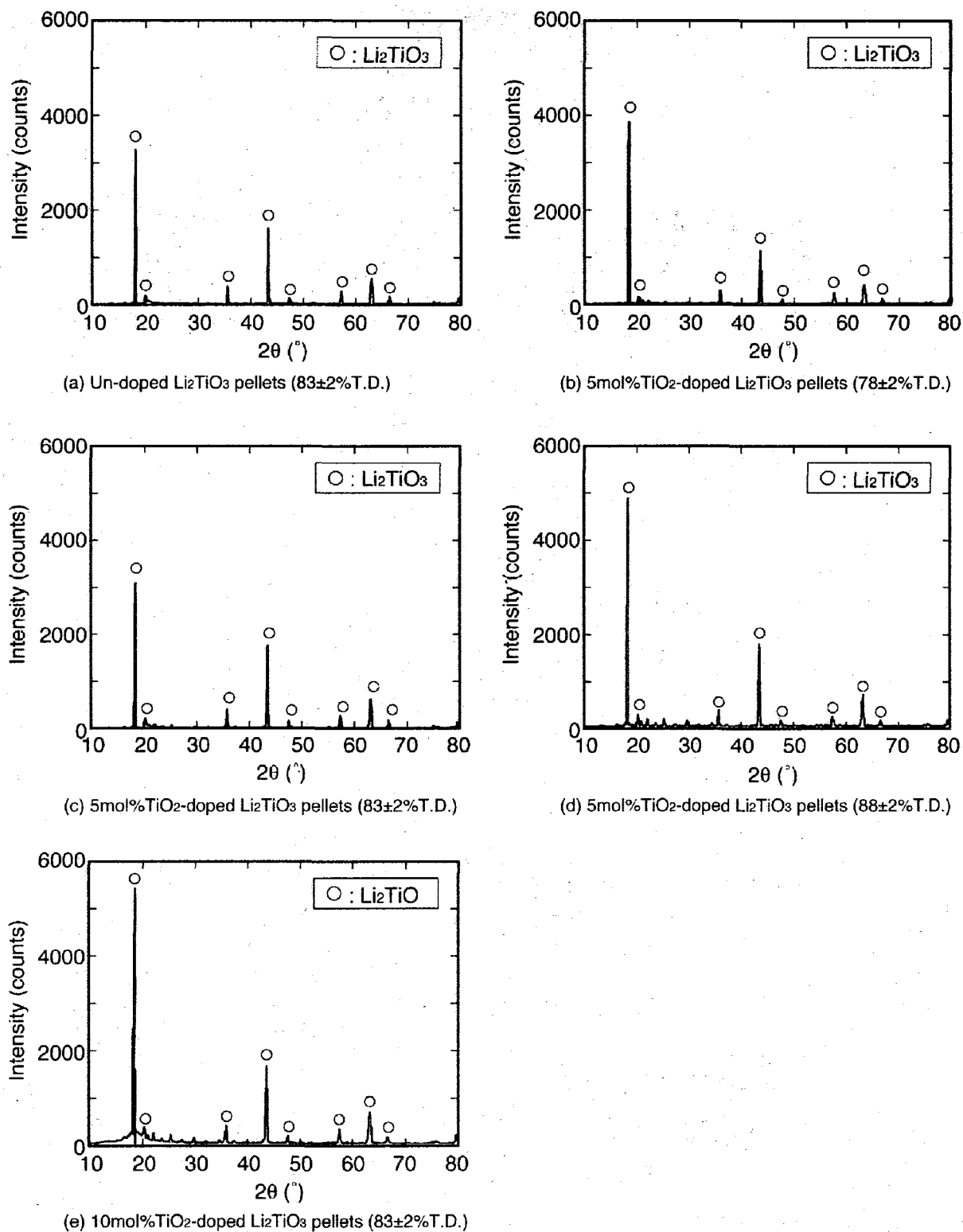


Fig.A-3 X-ray diffraction pattern of ^6Li -enriched Li_2TiO_3 and TiO_2 -doped ^6Li -enriched Li_2TiO_3 pellets.

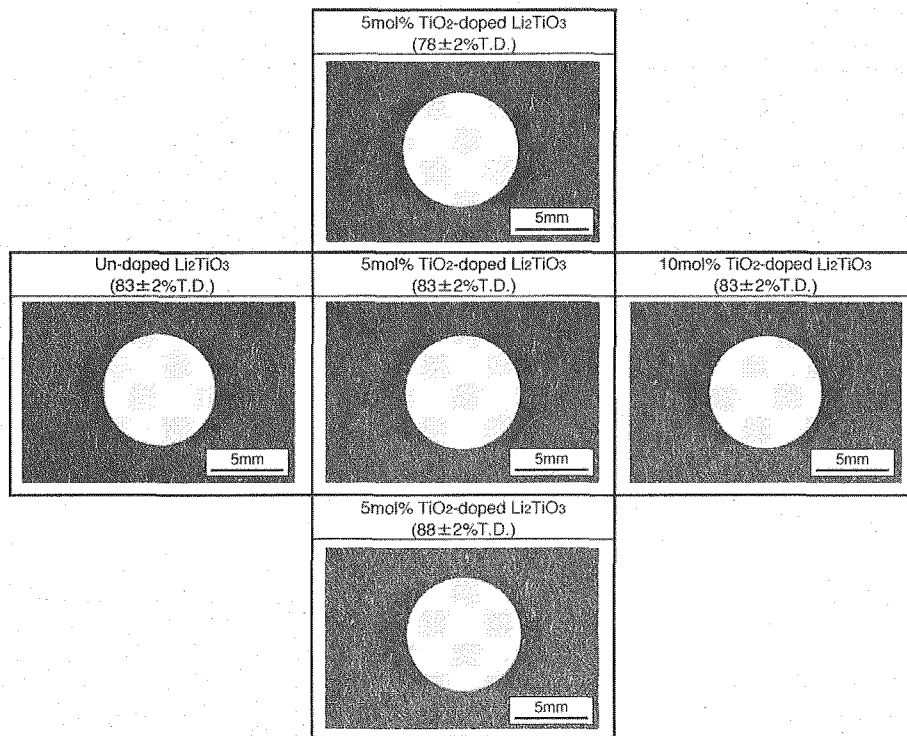


Fig. A-4 Photographs of ^6Li -enriched Li_2TiO_3 pebbles.

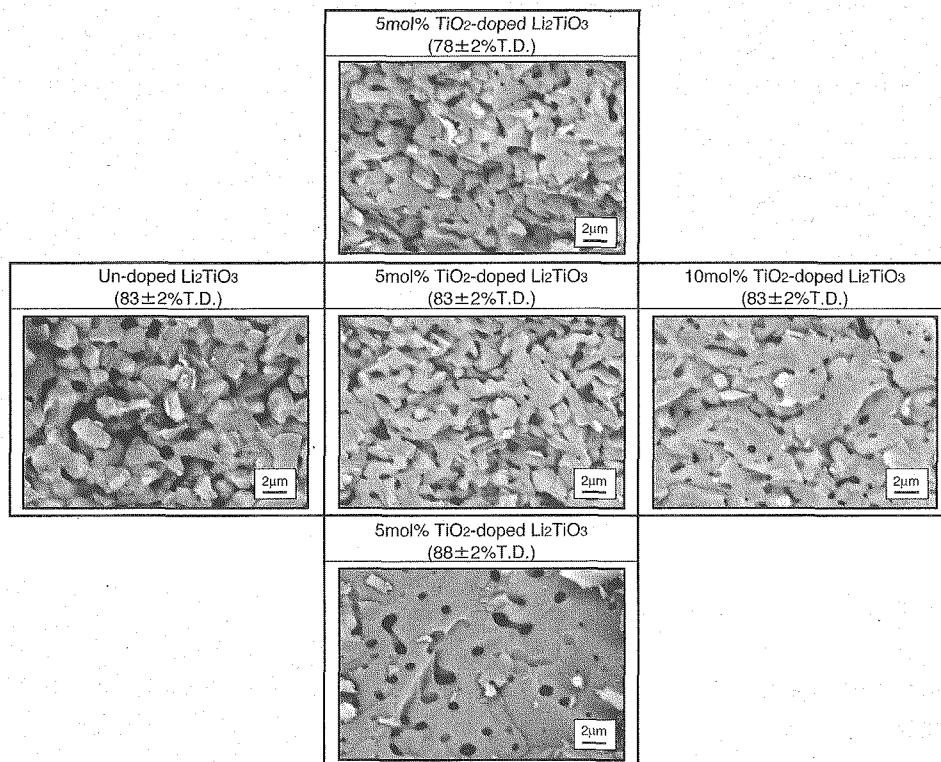


Fig.A-5 SEM photograph of ^6Li -enriched Li_2TiO_3 pellets.

国際単位系 (SI)

表 1. SI 基本単位

基本量	SI 基本単位	
	名称	記号
長さ	メートル	m
質量	キログラム	kg
時間	秒	s
電流	アンペア	A
熱力学温度	ケルビン	K
物質の量	モル	mol
光の度	カンデラ	cd

表 2. 基本単位を用いて表されるSI組立単位の例

組立量	SI 基本単位	
	名称	記号
面積	平方メートル	m ²
体積	立方メートル	m ³
速度	メートル毎秒	m/s
加速度	メートル毎秒毎秒	m/s ²
波数	毎メートル	m ⁻¹
密度 (質量密度)	キログラム毎立方メートル	kg/m ³
質量体積 (比体積)	立方メートル毎キログラム	m ³ /kg
電流密度	アンペア毎平方メートル	A/m ²
磁界の強さ	アンペア毎メートル	A/m
(物質量の) 濃度	モル毎立方メートル	mol/m ³
輝度	カンデラ毎平方メートル	cd/m ²
屈折率	(数の) 1	1

表 5. SI 接頭語

乗数	接頭語	記号	乗数	接頭語	記号
10 ²⁴	ヨタ	Y	10 ⁻¹	デシ	d
10 ²¹	ゼタ	Z	10 ⁻²	センチ	c
10 ¹⁸	エクサ	E	10 ⁻³	ミリ	m
10 ¹⁵	ペタ	P	10 ⁻⁶	マイクロ	μ
10 ¹²	テラ	T	10 ⁻⁹	ナノ	n
10 ⁹	ギガ	G	10 ⁻¹²	ピコ	p
10 ⁶	メガ	M	10 ⁻¹⁵	フェムト	f
10 ³	キロ	k	10 ⁻¹⁸	アト	a
10 ²	ヘクト	h	10 ⁻²¹	ゼプト	z
10 ¹	デカ	da	10 ⁻²⁴	ヨクト	y

表 3. 固有の名称とその独自の記号で表されるSI組立単位

組立量	SI 組立単位			
	名称	記号	他のSI単位による表し方	SI基本単位による表し方
平面角	ラジアン ^(a)	rad		m・m ⁻¹ =1 ^(b)
立体角	ステラジアン ^(a)	sr ^(c)		m ² ・m ⁻² =1 ^(b)
周波数	ヘルツ	Hz		s ⁻¹
力	ニュートン	N		m・kg・s ⁻²
圧力, 応力	パスカル	Pa	N/m ²	m ⁻¹ ・kg・s ⁻²
エネルギー, 仕事, 熱量	ジュール	J	N・m	m ² ・kg・s ⁻²
工率, 放射束	ワット	W	J/s	m ² ・kg・s ⁻³
電荷, 電気量	クーロン	C		s・A
電位差 (電圧), 起電力	ボルト	V	W/A	m ² ・kg・s ⁻³ ・A ⁻¹
静電容量	ファラド	F	C/V	m ⁻² ・kg ⁻¹ ・s ⁴ ・A ²
電気抵抗	オーム	Ω	V/A	m ² ・kg・s ⁻³ ・A ⁻²
コンダクタンス	ジーメン	S	A/V	m ⁻² ・kg ⁻¹ ・s ³ ・A ²
磁束	ウェーバ	Wb	V・s	m ² ・kg・s ⁻² ・A ⁻¹
磁束密度	テスラ	T	Wb/m ²	kg・s ⁻² ・A ⁻¹
インダクタンス	ヘンリー	H	Wb/A	m ² ・kg・s ⁻² ・A ⁻²
セルシウス度 ^(d)	セルシウス度 ^(d)	°C		K
光学的密度	ルーメン	lm	cd・sr ^(c)	m ² ・m ⁻² ・cd=cd
照射度	ルクス	lx	lm/m ²	m ² ・m ⁻² ・cd=cd
(放射線核種の) 放射能	ベクレル	Bq		s ⁻¹
吸収線量, 質量エネルギー当量, カリマ線量当量, 周辺線量当量, 方向性線量当量, 個人線量当量, 組織線量当量	グレイ	Gy	J/kg	m ² ・s ⁻²
	シーベルト	Sv	J/kg	m ² ・s ⁻²

表 6. 国際単位系と併用されるが国際単位系に属さない単位

名称	記号	SI 単位による値
分	min	1 min=60s
時	h	1 h=60 min=3600 s
日	d	1 d=24 h=86400 s
度	°	1°=(π/180) rad
分	'	1'=(1/60)°=(π/10800) rad
秒	"	1"=(1/60)'=(π/648000) rad
リットル	l, L	1 l=1 dm ³ =10 ⁻³ m ³
トン	t	1 t=10 ³ kg
ネーパ	Np	1 Np=1
ベル	B	1 B=(1/2) ln10 (Np)

表 7. 国際単位系と併用されこれに属さない単位で SI 単位で表される数値が実験的に得られるもの

名称	記号	SI 単位であらわされる数値
電子ボルト	eV	1 eV=1.60217733 (49) × 10 ⁻¹⁹ J
統一原子質量単位	u	1 u=1.6605402 (10) × 10 ⁻²⁷ kg
天文単位	ua	1 ua=1.49597870691 (30) × 10 ¹¹ m

表 8. 国際単位系に属さないが国際単位系と併用されるその他の単位

名称	記号	SI 単位であらわされる数値
海里		1 海里=1852m
ノット		1 ノット=1 海里毎時=(1852/3600) m/s
アール	a	1 a=1 dam ² =10 ² m ²
ヘクタール	ha	1 ha=1 hm ² =10 ⁴ m ²
バール	bar	1 bar=0.1 MPa=100kPa=1000hPa=10 ⁵ Pa
オングストローム	Å	1 Å=0.1 nm=10 ⁻¹⁰ m
バイン	b	1 b=100fm ² =10 ⁻²⁸ m ²

表 4. 単位の中に固有の名称とその独自の記号を含むSI組立単位の例

組立量	SI 組立単位		
	名称	記号	SI 基本単位による表し方
粘力のモーメント	パスカル秒	Pa・s	m ⁻¹ ・kg・s ⁻¹
表面張力	ニュートンメートル	N・m	m ² ・kg・s ⁻²
角速度	ニュートン毎メートル	N/m	kg・s ⁻²
角加速度	ラジアン毎秒	rad/s	m・m ⁻¹ ・s ⁻¹ =s ⁻¹
熱流密度, 放射照度	ラジアン毎平方秒	rad/s ²	m・m ⁻¹ ・s ⁻² =s ⁻²
熱容量, エントロピー	ワット毎平方メートル	W/m ²	kg・s ⁻³
質量熱容量 (比熱容量), 質量エントロピー	ジュール毎平方メートル	J/m ²	m ² ・kg・s ⁻² ・K ⁻¹
質量エネルギー (比エネルギー)	ジュール毎キログラム	J/(kg・K)	m ² ・s ⁻² ・K ⁻¹
熱伝導率	ジュール毎キログラム	J/kg	m ² ・s ⁻² ・K ⁻¹
体積エネルギー	ワット毎メートル毎ケルビン	W/(m・K)	m・kg・s ⁻³ ・K ⁻¹
電界の強さ	ジュール毎立方メートル	J/m ³	m ⁻¹ ・kg・s ⁻²
体積電荷	ボルト毎メートル	V/m	m・kg・s ⁻³ ・A ⁻¹
電気変位	クーロン毎立方メートル	C/m ³	m ⁻³ ・s・A
誘電率	クーロン毎平方メートル	C/m ²	m ⁻² ・s・A
透磁率	ファラド毎メートル	F/m	m ⁻³ ・kg ⁻¹ ・s ⁴ ・A ²
モルエネルギー	ヘンリー毎メートル	H/m	m ² ・kg・s ⁻² ・A ⁻²
モルエントロピー, モル熱容量	ジュール毎モル	J/mol	m ² ・kg・s ⁻² ・K ⁻¹ ・mol ⁻¹
照射線量 (X線及びγ線)	ジュール毎モル毎ケルビン	J/(mol・K)	m ² ・kg・s ⁻² ・K ⁻¹ ・mol ⁻¹
吸収線量	クーロン毎キログラム	C/kg	kg ⁻¹ ・s・A
放射強度	グレイ毎秒	Gy/s	m ² ・s ⁻³
放射輝度	ワット毎ステラジアン	W/sr	m ⁴ ・m ⁻² ・kg・s ⁻³ =m ² ・kg・s ⁻³
	ワット毎平方メートル毎ステラジアン	W/(m ² ・sr)	m ² ・m ⁻² ・kg・s ⁻³ =kg・s ⁻³

表 9. 固有の名称を含むCGS組立単位

名称	記号	SI 単位であらわされる数値
エルグ	erg	1 erg=10 ⁻⁷ J
ダイン	dyn	1 dyn=10 ⁻⁵ N
ポズ	P	1 P=1 dyn・s/cm ² =0.1 Pa・s
ストークス	St	1 St=1 cm ² /s=10 ⁻⁴ m ² /s
ガウス	G	1 G=10 ⁻⁴ T
エルステッド	Oe	1 Oe=(1000/4π) A/m
マクスウェル	Mx	1 Mx=10 ⁻⁸ Wb
スチル	sb	1 sb=1 cd/cm ² =10 ⁴ cd/m ²
ホト	ph	1 ph=10 ⁴ lx
ガリ	Gal	1 Gal=1 cm/s ² =10 ⁻² m/s ²

表 10. 国際単位系に属さないその他の単位の例

名称	記号	SI 単位であらわされる数値
キュリー	Ci	1 Ci=3.7×10 ¹⁰ Bq
レントゲン	R	1 R=2.58×10 ⁻⁴ C/kg
ラド	rad	1 rad=1 cGy=10 ⁻² Gy
レム	rem	1 rem=1 cSv=10 ⁻² Sv
X線単位		1 X unit=1.002×10 ⁻⁴ nm
ガンマ	γ	1 γ=1 nT=10 ⁻⁹ T
ジャンスキー	Jy	1 Jy=10 ⁻²⁶ W・m ⁻² ・Hz ⁻¹
フェルミ		1 fermi=1 fm=10 ⁻¹⁵ m
メートル系カラット		1 metric carat=200 mg=2×10 ⁻⁴ kg
トル	Torr	1 Torr=(101 325/760) Pa
標準大気圧	atm	1 atm=101 325 Pa
カリ	cal	
ミクロン	μ	1 μ=1 μm=10 ⁻⁶ m