Acknowledgement

This work was supported by a grant from the Rehabilitation Services Administration, Department of H.E.W. (No. 23-P-57377/2-02).

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Received 12 June
and accepted 21 July 1978.

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Preparation of $ThSiO_4$ single crystals by a vapour phase reaction

Thorium silicate is the only ternary compound which exists in the ThO_2 and SiO_2 system [1, 2]. Thorium silicate single crystals were synthesized by the reaction of an aqueous solution of thorium chloride with pyrex glass [3]. The present paper reports a similar process for the preparation of $ThSiO_4$ crystals using a vapour phase reaction. In the study of chemical transport reactions of uranium and thorium oxides the author has prepared single crystals of $ThSiO_4$ using halogen gases [4]. When ThO_2 source material is sealed in a quartz tube with HCl or Cl_2 gas as a transport agent and placed in the hot zone, $ThSiO_4$ single crystals can be produced almost in the cold zone in the quartz tube. The crystal is about $1 \times 0.7 \times 0.5\ mm^3$ in size and is transparent with many faces. The possible reactions are considered as follows:

$$ThO_2(g) + 4HCl(g) = ThCl_4(g) + 2H_2O(g), \quad (1)$$

some parts of $ThCl_4(g)$ would react with the quartz tube,

$$ThCl_4(g) + SiO_2(s) = ThO_2(s) + SiCl_4(g), \quad (2)$$

and then the formation of $ThSiO_4$ crystals proceeds in the colder zone as,

TABLE I Preparation conditions of $ThSiO_4$ crystals

	ThO_2 + HCl	ThO_2 + Cl_2
Source temperature (° C)	1050	1050
Deposition temperature (° C)	950	950
Pressure of halogen (atm at 1000° C)	3.49	1.56
Transport time (h)	347	936
Transport rate ($mg\ h^{-1}$)	0.17	0.26
Crystal size (mm^3)	0.2 × 0.2 × 0.2	1 × 0.7 × 0.5

$$SiCl_4(g) + ThCl_4(g) + 4H_2O(g) = ThSiO_4(s) + 8HCl(g). \quad (3)$$

Evidence that $ThSiO_4$ crystals may be prepared using a vapour phase reaction (Equation 3) is due to the facts:

(1) $ThSiO_4$ crystals can be produced in the cold zone more readily than in intermediate or hot zones.

(2) The crystals produced are randomly distributed in the cold zone as is usual in the case of chemical transport reactions, and not uniformly deposited all over the tube, as is the coating produced by the direct reaction of $ThCl_4(g)$ with the quartz tube.

When the temperature at the cold zone is below

900° C, the crystals of $ThSiO_4$ are scarcely produced.

Table I shows the preparation conditions. When uranium dioxide co-exists with ThO_2 in the hot zone, the crystals of $ThSiO_4$ produced become green and transparent. Even in these cases the lattice constants of $ThSiO_4$ do not vary, so that the solubility of uranium in $ThSiO_4$ is deduced to be so small as to be only a degree of order of doping. Fig. 1 shows a crystal of $ThSiO_4$ doped with uranium which is green in colour. The lattice parameters of the $ThSiO_4$ produced were $a_0 = 7.090 \pm 0.004$ Å and $c_0 = 6.317 \pm 0.004$ Å

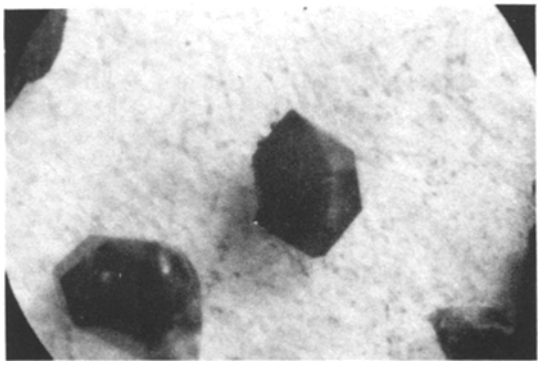

Figure 1 $ThSiO_4$ crystals doped with uranium (green in colour). Maximum length is about 1 mm.

Acknowledgements

The author would like to thank Professor K. Naito for valuable discussions and Mr Y. Nomura and Mr K. Ohta for experimental assistance. The FACOM 230-75 computer in Nagoya University was used for calculating the lattice constants.

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Received 9 June
and accepted 19 July 1978.

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The morphology of single crystals during compressive creep testing

Several investigators [1–3] reported that the change in morphology of single crystals undergoing compressive creep is a function of the loading system and that the shape of the creep curve is related to the morphology. Although their analyses were based on observations made on specimens at the completion of the creep test, the observations suggested that the type of morphology was directly related to the various stages of creep, i.e. primary, steady-state, etc. The present study was conducted to record the morphology changes of a specimen during a compressive creep test.

Time lapse photography was used to observe and record pictorially the specimen morphology as it deformed. After placing the specimen in the furnace, the telescope and camera were aligned so that the specimen could be clearly seen through the viewfinder of the camera. Focusing was done at room temperature by adjusting the objective lens of the telescope and the camera's focusing ring. At the temperatures (1000° C to 1300° C) employed during these experiments, the interior of the furnace essentially became a black body, making it impossible to distinguish the specimen from the background of the furnace. A blue glass filter was used to eliminate most of the light radiated by the hot furnace. To provide the contrast required for photography, a high intensity white light source of at least 300 W was used to illuminate the specimen. The blue filter, although filtering out most of the light created by the high temperature conditions, would allow enough light to pass through to enable the image of the specimen to be recorded. A Kodak Cine-Special 16 mm movie camera with double X negative film was used to record the specimen morphology. Generally, 12 frames per minute were exposed during the experiment.