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BRIEF COMMUNICATIONS

Neutron and X-Ray Diffraction Study on Polymorphism in Lithium Orthotantalate, Li_3TaO_4: Correction and Discussion

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In the paper with the above title, by M. Zocchi, M. Gatti, A. Santoro, and R. S. Roth (*1*), an incorrect statement was inadvertently made in the introduction concerning the polymorphic relations of the *disordered* rock salt phase of Li_3TaO_4. This phase, with a lattice parameter of $a = 4.203$ Å, was reported by Lapicky and Simanov (*2*) and by Pfeiffer (*3*). Blasse (*4*), however, was unable to prepare this modification. Martel and Roth (*5*) found an unquenchable phase transition at about 900°C. In Ref. (*1*), it is incorrectly stated that this intermediate temperature phase "quite likely is the same modification as that reported by Lapicky and Simanov (*2*) and by Pfeiffer (*3*)." This cannot be true, as it would mean that a completely reconstructive transition from a disordered to an ordered state occurred in fractions of a second below 900°C, on quenching.

The three phases reported by Martel and Roth (*5*) are probably the only *stable* polymorphs of Li_3TaO_4. However, in the preparation of specimens for neutron diffraction, several other *metastable* polymorphs were encountered. The apparently disordered rock salt phase with lattice parameter $a \sim 4.2$ Å was easily prepared by heating equimolar proportions of Li_2CO_3 and $LiTaO_3$ at 750°C for about 60 hr. The x-ray pattern of this phase has a *very* broad diffuse reflection from about 15° to 30° 2θ (Cu$K\alpha$ radiation), indicative of extreme disorder, and all of the cubic peaks are broad and relatively diffuse. Longer heat treatment at 750°C or shorter times at 800°C result in the formation of a phase with two extra reflections at 23.08° (3.85 Å) and 24.38° (3.648 Å) but with all the other peaks of the 4.2-Å cubic phase. Still further heat treatment (time or temperature) causes the equilibrium formtion of the β phase originally reported by Blasse. Once the β phase is formed, the 4.2-Å cubic phase cannot be obtained by any (known) heat treatment, indicating that this phase is only metastable. However, if the Li_3TaO_4 is quenched very quickly from above the melting point (observed to be between 1525 and 1550°C), the 4.2-Å cubic phase is observed with an x-ray pattern having very sharp α_1–α_2 peaks with an additional peak at 23.50° 2θ (~3.78 Å), indicating a doubled cell with $a = 8.434$ Å. If the liquid is not quenched quite as rapidly, a still further metastable phase

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results, of unknown unit cell size and symmetry.

Thus, the unquenchable phase observed by DTA which occurs between 900 and 1400°C (*5*) is only one of several uncharacterized forms of Li_3TaO_4 but must represent an ordered, stable modification. This phase should be studied by high-temperature X-ray diffraction in order to characterize the transition.

References

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5. L. C. Martel and R. S. Roth, *Bull. Amer. Ceram. Soc.* **60,** 376 (1981).