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Heat treatment of some metamict allanites

LIMA-DE-FARIA (1958, 1964) studied X-ray powder patterns of heat-treated allanites, the object in that work being to identify, by X-ray diffraction, metamict minerals that showed no discernible powder lines before heating. We have studied the effect of heat-treatment in air and vacuum on three metamict allanites, using X-ray and Mössbauer methods. The allanites were supplied by the Department of Mineralogy, British Museum (Natural History). The specimens were originally from North Carolina, U.S.A. (BM 1957, 302), Ontario, Canada (BM 1924, 314), and Arendal, Norway (BM 42769), and were designated as A1, A2, and A3 respectively. Autoradiographic measurements, using previously analysed zircons (Vance and Mackey, 1974) as standards, gave the respective equivalent uranium contents as 0·5, 0·5, and 0·2 wt %.

X-ray diffraction data were obtained photographically, using filtered Cu radiation and a camera of 35 mm diameter. Although the samples contained iron, fluorescence was not a problem. The X-ray samples were single-crystal fragments, 1 to 2 mm in size. Data were obtained from the edges. Some powder patterns were also obtained. ^{57}Fe Mössbauer spectra of powders were measured using an arrangement of spectrometers described by Window *et al.* (1974). The samples were heated in platinum crucibles and the duration of the anneals was one hour.

No Laue spots or powder lines were observed on stationary-crystal X-ray photographs obtained from each sample; accordingly, the samples were severely damaged. The samples gave very broad Mössbauer spectra, which were similar to those obtained by Dollase (1971) on damaged samples. After A1 was annealed in air at 400 °C, a diffuse Laue pattern was obtained and the pattern sharpened after annealing in air at progressively higher temperatures up to 900 °C. After annealing at 700 °C in air, rather broad lines were observed on powder photographs; the *d* spacings were 2·92, 2·15, 1·90, and 1·64 Å, in agreement with the results of Lima-de-Faria (1958) for a Greenland allanite annealed at 700 °C in air. The Mössbauer spectrum sharpened considerably after annealing in air at 700 °C and agreed with with spectra of similarly heated allanites (Dollase, 1971); annealing in air increased the Fe^{3+}/Fe^{2+} ratio, as found by Dollase (1971) and Remy *et al.* (1970).

In contrast, no Laue spots were observed on stationary-crystal X-ray photographs obtained from A2 and A3 after annealing at 500 °C in air; however a faint, rather diffuse powder pattern indexing as CeO_2 was observed on the photographs from A2. An anneal at 700 °C in air was required to produce the CeO_2 pattern for A3. No Laue spots were observed on stationary-crystal photographs until after annealing at 800 and 900 °C in air; the CeO_2 pattern sharpened as a result. The Mössbauer results agreed essentially with those obtained on A1.

Annealing each specimen in vacuum at $\leqslant 900$ °C gave similar Mössbauer results to those obtained after heating in air at the same temperature, except that the increase in the Fe^{3+}/Fe^{2+} ratio was only slight. There were, however, indications of the formation of a small amount of Fe_3O_4 after annealing in vacuum at 700 to 900 °C (see also Lima-de-Faria, 1964). No CeO_2 pattern was observed on X-ray photographs. Otherwise, the X-ray results were similar to those obtained after annealing in air.

Both air and vacuum heat-treatments at $\gtrsim 1000$ °C produced decomposition, as evidenced by the appearance of powder lines on stationary-crystal X-ray photographs. Decomposition was complete after annealing at 1200 °C. From X-ray and Mössbauer studies, Fe_2O_3 (see also Remy *et al.*, 1970) and CeO_2 were the first phases to form on decomposition in air, while Fe_3O_4 and CeO_2 were the first phases to form on decomposition in vacuum. In both cases, the X-ray pattern of the residual structure agreed fairly well with that expected from lessingite (Gay, 1957), as found by Lima-de-Faria (1964); no cubic phase with a 3·52 Å (Lima-de-Faria, 1964) was found in either case.

The appearance of polycrystalline oxide phases on annealing in air at relatively low temperatures ($\lesssim 700$ °C) seems to be a common feature of minerals that have been severely damaged by the decay of radioactive impurities. Although Lima-de-Faria (1958, 1964) did not observe this behaviour in allanites heated in air at < 1000 °C, he did observe oxide phases after annealing polymignites, euxenites, yttrotantalites, samarskites, and pyrochlores at 700 °C in air. It would seem from the present work that Lima-de-Faria (1958, 1964) did not observe CeO_2 formation in allanites heated at 700 °C in air because the specimens studied by him were insufficiently damaged (see also the case of zircon—Vance and Anderson, 1972; Vance, 1975). In the present work we have studied also a metamict samarskite from the Strangways Range, Northern Territory, Australia: from X-ray diffraction it was found that the cubic betafite-like phase (Viswanathiah *et al.*, 1971) appeared after annealing at only 400 °C.

The fundamental reason for the 'precipitation' of cubic oxide phases after annealing radiation-damaged minerals at relatively low temperatures remains obscure, but it must relate to the detailed forms of the distortions of the structures of the minerals after severe radiation damage. The volume change involved in the 'precipitation' would also seem to be important (Vance, 1974). A complicating factor for allanite is that the formation of CeO_2 is favoured by an oxidizing atmosphere, as found by Adams and Sharp (1970); this presumably means that Ce^{3+} is oxidized to Ce^{4+}.

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Sapphirine in the Sittampundi Complex, India: A discussion

JANARDHANAN and Leake (1974) have described a plagioclase-rich rock from the Sittampundi Complex that has the assemblage: plagioclase+gedrite+colourless clinoamphibole+phlogopite+sillimanite+chrome-spinel+corundum+sapphirine. We have inadvertently synthesized sapphirine[1] as a product of incongruent melting of plagioclase during experimental studies of deformation of an anorthosite (plagioclase composition An 78–80, with a trace of chlorite) using talc as the confining medium, and feel this may have some bearing on the genesis of the rock described by Janardhan and Leake.

The experiments were carried out in a solid-medium deformation apparatus of the type described by Griggs (1967). A cylindrical sample of anorthosite 19 mm in length and 6 mm in diameter was surrounded by a 2 mm thick sleeve of talc within a thin graphite tube furnace. The furnace was enclosed in a 7 mm thick talc jacket through which the confining pressure was applied. In these experiments the temperature is at a maximum in the centre of the sample and decreases towards both ends due to conductive losses through the alumina end pieces. During uniaxial deformation at 850 °C and 10 kb confining pressure, the talc sleeve dehydrated yielding a vapour phase that must have been rich in Mg and Si. Plagioclase in the maximum-temperature region adjacent to the dehydrated talc melted incongruently to sapphirine+liquid due