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ANNEALING CHARACTERISTICS OF METAMICT GADOLINITE FROM RODE RANCH TEXAS

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ABSTRACT

Chemically analyzed metamict gadolinite was heated in air at varied temperatures and time intervals. At 1080°C gadolinite was well crystallized by 1 hour. At 880°C samples showed a lesser but constant level of crystallinity. At 780°C and 730° the gamma phase reported at 1300° by Lima-de-Faria (1964) was predominant, but it remained poorly developed after heating for several days. At 680° no recrystallization was detected after one week heating. DTA showed a large exotherm at 810° and smaller exotherms at 850° and 900°. The degree of recrystallization, determined by three X-ray diffractogram peak areas, was influenced more by temperature than by heating time.

INTRODUCTION

Completely metamict gadolinite (having no X-ray diffraction pattern) will recrystallize, giving the X-ray diffractogram of crystalline gadolinite. Problems arise, however, in obtaining consistent results after annealing metamict minerals prior to X-ray analysis if (1) other metamict impurities are present, (2) polymorphs occur, or (3) insufficient or excess temperature is applied during the annealing treatment. The present study describes in detail the annealing character of a chemically analyzed gadolinite; hopefully the data can be used for optimum recrystallization of metamict gadolinite specimens.

Standardized X-ray identification of a number of metamict minerals is considered by Lima-de-Faria (1964), who heated gadolinite in air and nitrogen at fixed conditions of 700°C for 3 hours, 1000° for 1 hour, and

1300° for 1 hour. In addition to gadolinite lines, a gamma phase of unknown nature was observed in 7 of 8 specimens after annealing at 1300° in air. The gamma phase was observed at lower temperatures in one sample only. Material for the current study was hand picked from a specimen of chemically analyzed gadolinite (Table 1) from the Rode Ranch pegmatite, described by Ehlmann, Walper, and Williams (1964).

Procedure

A sample of approximately 5 grams of material for each run was ground to pass a 150 mesh screen. The sample was put in a platinum crucible and suspended for a designated period of time in a preheated, platinum wound, vertical-core furnace. After removal from the furnace, the sample was reground and was scanned by an X-ray diffractometer at $2°2\theta$

Table 1. Chemical Analysis of Rode Ranch Gadolinite
(Weight Percent)

SiO_2	23.45	Y_2O_3	29.11
Al_2O_3	1.73	La_2O_3	0.30
Fe_2O_3	0.64	Ce_2O_3	1.57
FeO	11.66	Pr_2O_3	0.40
MnO	0.36	Nd_2O_3	3.00
MgO	0.21	Sm_2O_3	2.66
CaO	0.51	Eu_2O_3	0.05
BeO	9.25	Gd_2O_3	4.01
$(RE)_2O_3$	51.21	Tb_2O_3	0.75
ThO_2	0.45	Dy_2O_3	4.76
$H_2O(-)$	0.01	Ho_2O_3	0.75
$H_2O(+)$	0.49	Er_2O_3	1.90
		Tm_2O_3	0.35
Total:	99.97	Yb_2O_3	1.35
		Lu_2O_3	0.25
		Total R.E:	51.21

Analysts: B. Bruun and S. Bergstøl, Geologisk Museum, Oslo, Norway.

per minute for qualitative analysis. The samples then were stored until quantitative X-ray analysis of the degree of crystallization could be performed on all samples treated at a particular temperature. Ratio of integrated peak intensities, read with a planimeter, was taken as an indication of the crystallinity of the annealed sample. This ratio was calculated by dividing the intensity of a peak from the trial sample by the intensity of the same peak on gadolinite "fully" annealed at 1080°C for 48 hours. The lines considered were 4.72, 2.82 and 2.55 Angstroms.

Results of Heating

1080° Series (1, 2, 4, 6, 8, 48 hours): X-ray diffractograms showed sharp patterns with the same intensities for all samples. Figure 1 includes the diffractogram of the 4 hour sample. The lines include those of ASTM

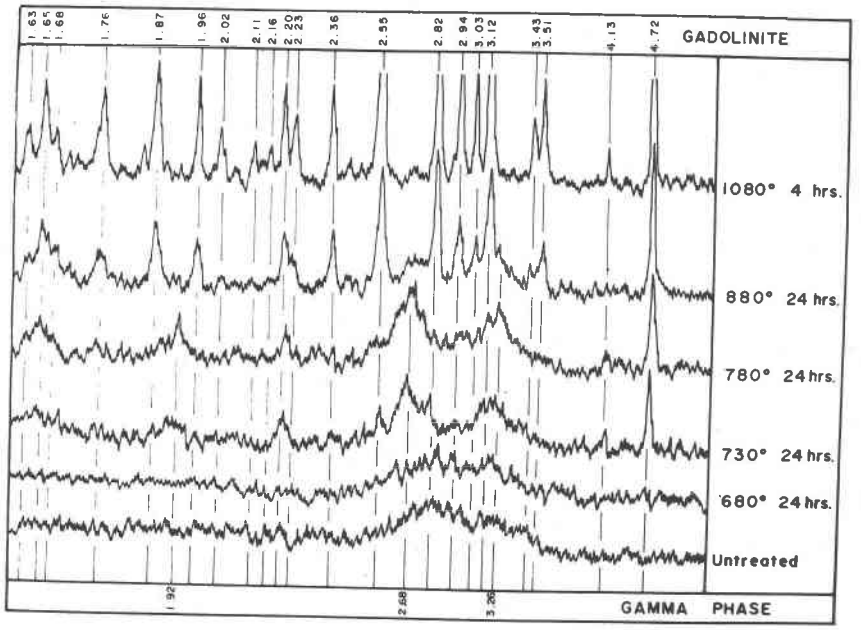

FIG. 1. X-ray Diffractograms of Untreated and Annealed Metamict Gadolinite from the Rode Ranch Pegmatite. The gamma phase is from Lima-de-Faria (1964). CuKα radiation.

Data Card #8–190 and correspond closely to the gadolinite lines listed by Lima-de-Faria (1964). There is no indication of the gamma phase.

880° Series (1/2, 1, 2, 4, 6, 8, 10, 12, 16, 18, 22, 24, 48 hours): By 1/2 hour recrystallization reached a maximum, which the 4.72Å line showed to be approximately 85 percent of the 1080° "standard," while the 2.82 Å and 2.55 Å lines showed approximately 50 percent intensities. Figure 1 shows that most of the gadolinite lines and only a slight indication of the 2.68 Å gamma phase line are present. Intensity of this gamma phase line decreased somewhat with increased heating time, but it was weak in all samples.

780° Series (1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22, 24, 48, 72 hours): Most of the gadolinite lines did not develop. The 4.72 Å gadolinite line increased in intensity from 30 percent of the standard after 1 hour to about 70 percent. As shown in Figure 1, the strongest lines belong to the gamma phase of Lima-de-Faria (1964). All samples in this series are similar.

730° Series (4, 15, 24, 66, 168, 336 hours): As shown in Figure 1, the lines from both phases (except the 4.72 Å gadolinite line) are poorly developed. The 4.72 Å line reached a maximum intensity of 70 percent after 24 hours.

680° Series (1, 2, 4, 9, 16, 24, 41, 72, 168 hours): As Figure 1 shows, diffractograms indicate no recrystallization and little change from the unheated gadolinite.

Results of DTA

Differential thermal analysis of this metamict gadolinite (heated at 12.5°C/min.) shows a large exotherm at 810°C grading into smaller exotherms at 850° and 900°, which correspond with recrystallization as shown by the heating experiments. A broad subdued exotherm occurs at 275°–350°; this may represent some increase in ordering of the metamict gadolinite although not sufficient to be shown by X ray. An endotherm occurs at 760°; it may be related to the appearance of the gamma phase prior to recrystallization of the gadolinite.

Conclusions

Recrystallization of gadolinite depends on temperature rather than time of heating. Heating of one hour at approximately 1100° provides well crystallized material for X-ray analysis. The nature of the gamma phase remains uncertain. Here it seems transitional and exists between roughly 700° and 800°C. This conflicts with the study of Lima-de-Faria (1964).

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