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UNITED STATES
DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY
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Mr. James E. Reeves
Assistant Manager for Test Operations
Albuquerque Operations Office
U. S. Atomic Energy Commission
P. O. Box 5400
Albuquerque, New Mexico

Dear Mr. Reeves:

Transmitted herewith are ten copies of TEI-729, "The action of heat and of superheated steam on the tuff of the Oak Spring formation," by George W. Morey, August 1958.

This report is part of our Nevada Test Site project. We plan to incorporate the information in TEI-729 with that in other reports on this project for publication by the Geological Survey.

Sincerely yours,

for W. H. Bradley
Chief Geologist

UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

THE ACTION OF HEAT AND OF SUPERHEATED STEAM ON THE TUFF OF THE OAK SPRING FORMATION*

By

George W. Morey

August 1958

Trace Elements Investigations Report 729

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*This report concerns work done on behalf of Albuquerque Operations Office, U. S. Atomic Energy Commission.

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THE ACTION OF HEAT AND OF SUPERHEATED STEAM ON TUFF OF THE OAK SPRING FORMATION

By George W. Morey

ABSTRACT

Samples of tuff of the Oak Spring formation heated in air at different temperatures were examined with the petrographic microscope and by X-ray diffraction methods. The changes in the heulandite, which makes up the greater part of the tuff, can be used to estimate the temperature to which the tuff had been heated. The tuff becomes fluid enough to begin to flow under its own weight at about 1,200° C in air, but even at higher temperatures the molten material has extremely high viscosity.

Samples of the tuff were heated in superheated steam at high pressures, and from these experiments it is concluded that recrystallization of the heulandite with formation of plagioclase feldspar is indicative of the presence of steam at high temperature and pressure during recrystallization.

INTRODUCTION

The underground nuclear explosion of September 19, 1957 took place in a rhyolitic tuff of the Oak Spring formation of Tertiary age. In the specimen used for the experiments described below the originally glassy shards have been completely altered, chiefly to the zeolite mineral heulandite [$(Ca,Na_2)O \cdot Al_2O_3 \cdot 6SiO_2 \cdot 5H_2O$]. Heulandite makes up about 70 percent of the rock specimen. The remainder is about 20 percent quartz, many of bipyramidal habit indicating that they were formed as high-quartz above 575° C, and

10 percent of K feldspar, plagioclase, hornblende, magnetite, and biotite (Hansen and Lemke, 1958). A considerable amount of water is also present. Some of this is combined in the heulandite, which ideally contains 15 percent water. If the tuff is 70 percent heulandite, it should have a minimum water content of 10 percent. Actual analyses of tuff range from 10 to 29 percent water, so the quantity of water in the tuff actually subject to the heating effects of the nuclear explosion is uncertain.

The author takes pleasure in acknowledging the assistance of W. F. Outerbridge in the petrographic examinations and of Daphne R. Ross in the X-ray diffraction studies. The work herein reported is part of a program being conducted by the U. S. Geological Survey on behalf of Albuquerque Operations Office, U. S. Atomic Energy Commission.

EXPERIMENTS IN AIR

Samples of tuff taken from the explosion chamber prior to the shot, and identified by the number G57-9-1, were powdered, placed in small crucibles hanging in an automatically controlled quenching furnace, and heated in air. After heating, the samples were removed and examined with the petrographic microscope and X-ray diffractometer. The experimental conditions and results of these experiments are shown in table 1.

The author estimates, from the chemical composition of the material and the melting relations of pertinent systems which have already been studied at the Geophysical Laboratory, Carnegie Institution of Washington, that the temperature of completion of the melting process is about 900° C. This estimate presupposes that the material was originally crystalline and that equilibrium was reached. The last crystalline phase to disappear would be

Table 1.--Effect of heating tuff of the Oak Spring formation in air.

Temperature (C)	Time	Condition
360	4 days	X-ray pattern shows heulandite altered to unidentified. Other minerals unchanged.
500	18 hrs	Heulandite has decomposed to glass. Other minerals unchanged.
760	42 hrs	Do.
1,032	42 hrs	Not sintered. Heulandite has decomposed to glass. Other minerals unchanged.
1,150	8 days	Partly sintered; heulandite decomposed to bubbly glass. Other minerals unchanged.
1,160	4 hrs	Thoroughly sintered and cemented together by bubbly glass. Other minerals unchanged.
1,191	6 days	Melted, but phenocrysts of quartz and K feldspar are not altered. In addition to the bubbly glass formed from heulandite, there is a clear glass probably formed from the plagioclase.
1,205	5 days	Melted to bubbly glass with some phenocrysts of quartz and K feldspar remaining metastably.

silica in the form of quartz or tridymite. The tuff originally, however, was largely glass, so the melting process was near completion, but the phases present, quartz and feldspar, are as would be expected. However, equilibrium in mixtures of this general composition was reached very slowly, as is shown in table 1. At 1,032° C the heulandite had decomposed to a glass, but the material was still so viscous that the powder did not sinter together in 42 hours. At 1,150° C sintering had begun, and at 1,191° C the material was thoroughly melted together. Two types of glass could be distinguished, one a bubbly glass formed by decomposition of the heulandite, and the other a clear glass chiefly formed by the melting of the plagioclase feldspar. The behavior of these materials on heating is consistent with the known viscosity of silicate melts. At 1,200° C an ordinary soft glass like window glass will have a viscosity of 10^3 poises; molten albite about 10^7 poises and molten orthoclase, 10^8 poises.

When tuff is heated at lower temperatures (500° and 760° C) about the only change is the driving off of the combined water in heulandite, resulting in the formation of a bubbly glass, and the condition (table 1) of the heulandite is evidently diagnostic of the temperatures to which the tuff had been subjected. To secure further information on this, samples of a well-crystallized heulandite from Iceland were heated in air at the temperatures and for the time given in table 2, and the products studied by X-ray diffraction. At 199° C the heulandite pattern remained unaltered. At 290° C with short exposures the X-ray pattern obtained was that of heulandite, but with slightly altered spacings. However, when heated for

Table 2.--Effect of heating heulandite in air.

Temperature (C)	Time (hrs)	Condition
199	17	Heulandite remained unaltered.
	64	Do.
290	1	X-ray pattern was essentially that of heulandite, with slightly altered spacings.
	64	X-ray pattern showed that the heulandite had completely altered to an unidentified compound.
360	1	Same unidentified pattern.
	4	Do.
	113	Do.
505	3	Noncrystalline, except for a trace of quartz.
	20	Do.

64 hours, the tuff yielded a new X-ray pattern (table 3) which did not match those of any minerals in the files of the U. S. Geological Survey. This unidentified phase persisted even after 113 hours heating at 360° C. This was the same pattern obtained by heating tuff to 360° C for 4 days (referred to as "unidentified" in table 1).

The alteration of the heulandite evidently provides a clue to the temperature at which the tuff was heated as the result of the heat given off by the explosion and it is planned to make X-ray diffraction studies of samples obtained in the survey of the region of the collapsed detonation cavity.

EXPERIMENTS IN SUPERHEATED STEAM AT HIGH PRESSURE

Inasmuch as a large amount of steam was formed as a result of the explosion (Johnson and others, 1958) an attempt was made to find the effect of superheated steam on tuff at high pressures. The apparatus used was that described by Morey and Hesselgesser (1952), in which a closed pressure vessel of Inconel X was heated in a furnace with automatic temperature control. About a half-gram sample of the powdered tuff, identified by the number G57-9-1, was placed in a platinum crucible with a little water, put into the bomb, and heated to the desired temperature. The pump was then started, and water pumped into the hot bomb with an automatically controlled pump until the desired pressure was reached. The results are shown in table 4.

The lowest temperature chosen was 500° C. In 3 hours at 5,000 psi, the heulandite had become almost entirely noncrystalline, just as in the runs in air, but a new unidentified pattern had begun to develop which had some of the strong lines of the plagioclase feldspars. In 96 hours at

Table 3.--X-ray pattern of the unidentified derivative of heulandite which was obtained by heating heulandite from Iceland to 290° C for 64 hours, and heulandite in tuff of the Oak Spring formation to 360° C for 4 days.1/

X-ray powder data for unidentified film no. 11779, CuKα radiation, λ = 1.5418 A, Ni filter, cutoff = 11 A, camera diameter = 114.59 mm

d (A)	I
8.3	100
7.6	25
6.7	35
5.6	25
5.2	35
4.96	35
4.27	9
4.13	9
3.90	6
3.80	35
3.65	50
3.52	9
3.36	71
3.27	13
3.13	6
2.93	3
2.91	3
2.82	18
2.76	4
2.69	6
2.56	6
2.24	3
2.19	2

1/ Analyst, Daphne R. Ross, U. S. Geological Survey.

Table 4.--The effect of superheated steam at high pressure on the tuff of the Oak Spring formation.

Temperature (C)	Pressure (psi)	Time (hrs)	Condition
500	5,000	3	Heulandite has become almost entirely noncrystalline with some plagioclase feldspar present in it. Phenocrysts not appreciably altered.
500	6,500	96	Heulandite has recrystallized to plagioclase feldspar. Phenocrysts not appreciably altered.
600	15,000	17	Heulandite recrystallized to plagioclase. Phenocrysts not appreciably altered.
650	15,000 30,000	18 2	Heulandite has recrystallized to a plagioclase feldspar, probably oligoclase. Phenocrysts not appreciably altered.

6,500 psi the heulandite had changed almost completely to plagioclase, but the crystals were still too fine grained for the plagioclase to be identified. At 600° C and 15,000 psi the plagioclase lines were better developed. Finally, a run was made at 650° C and 15,000 psi for 18 hours, then the pressure was increased to 30,000 psi for two hours. In this run the feldspar was tentatively identified as an oligoclase, a plagioclase in the albite-anorthite series containing from 10 to 30 percent anorthite. It is probable that this is the feldspar formed in all of the runs at lower temperature and pressure. It is noteworthy that a glass of the composition of oligoclase will not crystallize even in many months when heated dry at 500° C. In all of the above runs the phenocrysts of quartz, K feldspar, and plagioclase apparently remained unaltered.

A sample of the Iceland heulandite heated for 3 hours at 500° C and 15,000 psi steam pressure contained some noncrystalline material, plagioclase, and some quartz.

It is evident that the change of heulandite to feldspar is indicative of an exposure to superheated steam, and any study of the tuff collected from the collapsed explosion chamber should include an X-ray diffraction study of the material with special attention to the changes in the heulandite. Any change to plagioclase would be indicative of exposure to superheated steam.

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