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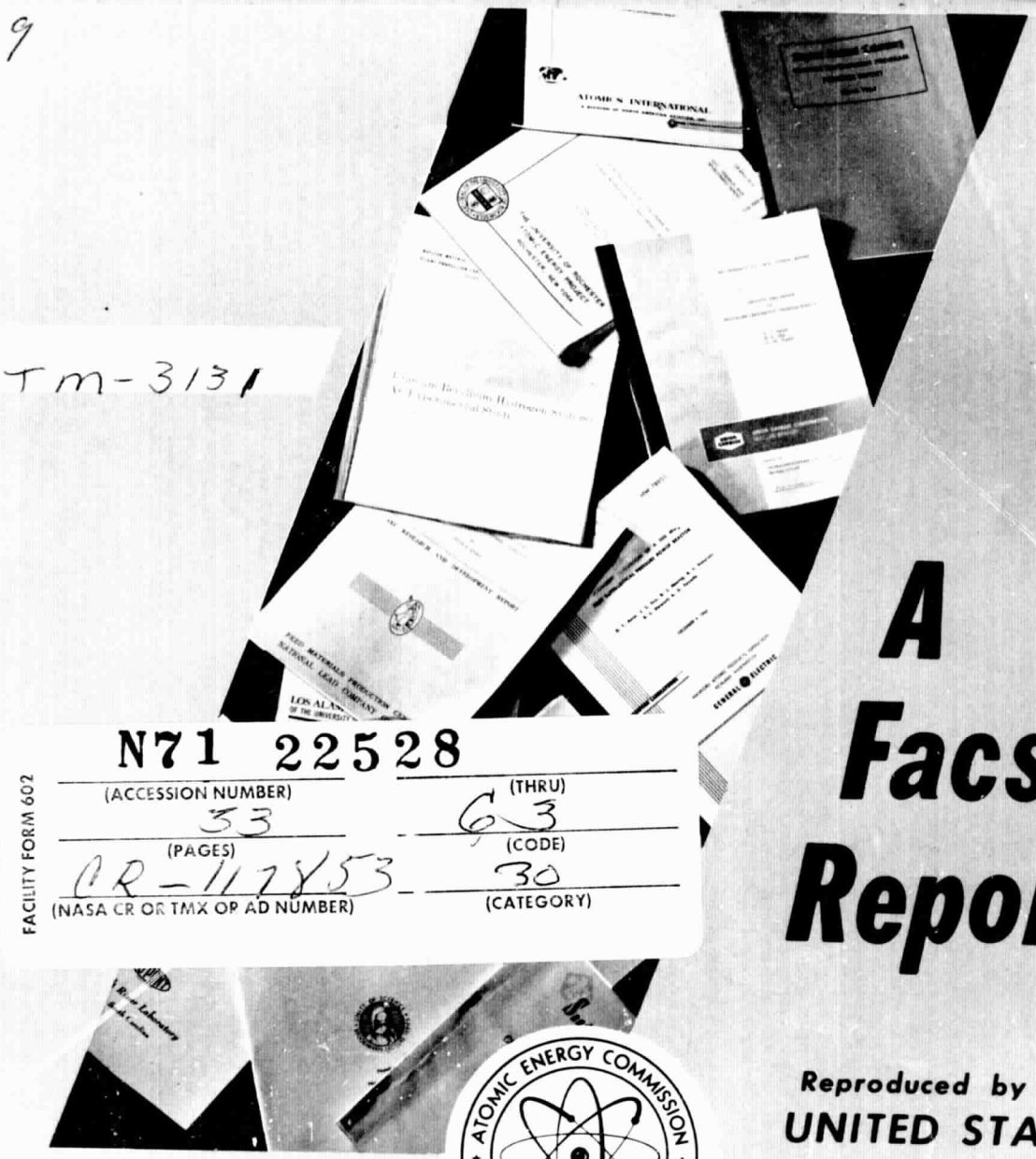

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LUNAR SAMPLE ANALYSIS PROGRAM

RESEARCH ON STIMULATED EXOELECTRON EMISSION FROM LUNAR MATERIALS

Technical Progress Report for Proposal No. 43-035-(002)

by

R. B. Gammage and Klaus Becker

August 28, 1970

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OCTOBER 1970

by

Health Physics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee

for

National Aeronautics and Space Administration
Manned Spacecraft Center
Lunar Receiving Laboratory
Houston, Texas

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Research on Stimulated Exoelectron Emission from Lunar Materials*

(Progress Report)

R. B. Gammage and Klaus Becker

ABSTRACT

Preliminary results on the thermally stimulated exoelectron emission (TSEE) from Apollo 12 fines and rocks show little emission from virgin materials, but intense TSEE at various temperatures between ∿130 and ∿550°C can be induced by (a) oxidation of the surface, (b) adsorption of O_2, and perhaps (c) the release of trapped solar wind. The TSEE characteristics of differently treated samples are a sensitive indicator of changes occurring on the surface and have, therefore, been studied in some detail. Further experimental plans are outlined.

Descriptors:

A. General: exoelectrons
surface properties
adsorption
oxidation
solar wind

B. Specific: exoelectrons, thermal stimulation
exoelectrons, optical stimulation
adsorption of oxygen
oxidation of iron
solar-wind de-trapping
atmospheric effects

*Research sponsored by the U. S. Atomic Energy Commission under contract with the Union Carbide Corporation.

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Summary

Preliminary results on the thermally stimulated exoelectron emission (TSEE) characteristics of several types of fines and rock samples from Apollo 12 show that only little, if any, TSEE occurs from the surface of "virgin" materials. A high TSEE rate of irradiated samples at various temperatures between ∿130 and ∿550°C can be induced by the superimposed effects of (a) oxidation of surface metals (Fe), (b) adsorption of O_2, and/or perhaps (c) the release of trapped solar wind. TSEE apparently is a highly sensitive tool for studying subtle changes which occur in very thin surface layers of lunar materials. Some further experiments are outlined.

1. Introduction

Natural and/or artificial radiation-induced thermoluminescence in Apollo 11 fines and crystalline rocks has been reported by several investigators [1-4], but failed to produce substantial new information on the radiation or thermal history of lunar surface materials mainly due to its rapid fading. It was initially thought [5] that the related, more direct approach in investigating electrons which are released from trapping centers during thermal or optical stimulation, exoelectron emission†, would provide a better tool because TSEE is a more widespread phenomenon and permits the studies of deeper, more stable trapping levels.

Initial attempts to find any TSEE in "virgin" (untreated and unirradiated) lunar surface fines failed. Further studies proceeded along two lines aimed at

†For a review on principles and applications of stimulated exoelectron emission, see K. Becker, IAEA Atomic Energy Review 8, 173, 1970.

(1) either uncovering natural TSEE activity in samples which had not been subject to the annealing influence of intense light or high temperature, or discovering the reasons for its suppression; and

(2) treating the lunar materials so as to induce changes in the artificial TSEE properties.

The latter experiments aim to identify the sources of the exoelectron emission. The induction of TSEE activity by treatments on earth has more than passing interest; lunar materials are in an unstable condition and are prone to attack by atmospheric gases. The extreme sensitivity of exoelectron emission to changing surface properties permits one to detect and follow degradation of, and reactions within, the lunar material as a function of temperature and ambient atmosphere. The value of such information for other studies is self-evident.

2. Search for Natural TSEE from Virgin Samples

In samples of about 10 mg each of surface fines No. 12070,18 and 12033,46 as well as in a core tube sample No. 12028,105 (parent 12028-32), only a very slight increase of the background amounting to a few hundred counts, or no increase at all has been observed during the first heating of the untreated material *. After exposure of the virgin material to radiation doses of up to 10^6 rad of γ rays, only a small increase in the count rate, principally around 450°, has been recorded. This result indicates that thermal or optical annealing at the lunar surface, or since, cannot be the only reason for the absence of TSEE.

Suspecting that solar wind buried in the surface might interfere with TSEE, material from the inside of a crystalline rock 12065,85† was

* For a description of the instrumentation and experimental techniques, see ref. 5

† Fragment of large (2109 g) pigeonite porphyry rock (plagioclase, pigeonite, and ilmenite) from totebag sample.

studied. A sample scraped from one of the cut faces exhibited no TSEE. Localized frictional heating occurring during the rock cutting could, however, already have released any trapped exoelectrons. Indeed some optical stimulation experiments in the sawed, unirradiated surface which were performed with Prof. Kramer's equipment in Braunschweig, Germany, did not yield any exoelectrons. Therefore, the rock fragment was fractured and the material so exposed was examined. This produced the first clear evidence of natural TSEE. A 1.2 mg portion of scrapings gave 1800 counts above the background with concentrations between 200 - 300°, 400 - 450° and 500 - 550°C. The readout was made within an hour of the fracture. Later readouts gave lower counts, perhaps due to the quenching effect of adsorbed atmospheric gases.

In another experiment, a larger amount (~6 mg) of scrapings from a fresh surface was heated at a faster rate (~6°C/sec.) in a more sensitive TSEE counter at NRL, Washington. Unfortunately, this reader permits heating only up to about 500°C. A total of 6000 counts was obtained, with a small peak (~400 counts) around 150°C, a higher peak around 450°C, and a strong indication of another peak around 500 - 550°C. More detailed experiments are planned for the near future.

Grounding of rock particles (12065,85) in a glazed mortar did not induce any detectable tribo-TSEE. Exposure of fines for 6 hours to a 10 W mercury vapor lamp also did not induce any signals in the rock powder but a small peak around 580°C appeared in virgin fines 12033,64. Exposure of a preirradiated fines sample for several hours to bright sunlight reduced, incidentally, the TSEE substantially due to optical annealing.

In summary, it seems that the detection of natural exoelectrons from virgin lunar materials is not to become a routinely easy matter. In future experiments we intend to remove the outer, solar wind containing layer from the lunar fines by chemical etching. We will also fracture rocks in a protective gas atmosphere to avoid disturbing atmospheric influences.

3. Artificial Inducement of Exoelectron Activity

Oxidation and reduction, adsorbed atmospheric gases and probably buried solar wind all appear to influence the TSEE characteristics of lunar materials. Most of our recent efforts have been directed toward identifying their effects.

A. Heat treatment

Repeated heating of the fines in the counting gas (99.05% He, 0.95% isobutane) was sufficient to impart exoelectron activity to the fines; the effects of consecutive heat cycles and irradiations upon fines 12033,46 are shown in Fig. 1. Fines No. 12070,18 were heated in air for periods of up to 24 hours (Fig. 2). A core tube sample (Fig. 3) exhibited a similar behavior. It may be noted that the TSEE curves are quite similar; small differences are probably caused by the varying composition*. In further experiments, the TSEE of some separated mineral constituents will be investigated.

Fig. 4 records the total TSEE between 75 and 600°C as a function of the duration of the heat treatment. Peaks grow at different rates resulting in changes in the peak ratios. This leads to a different slope in the TSEE versus dose curve (Fig. 5) for the low-temperature and the high-temperature peaks. The total TSEE is not a linear function of dose and no saturation is observed up to 10^6 rad.

*According to NASA-SP-235, p. 200, sample 12033 is notably different from other samples and is tentatively considered a crystal-vitric ash.

The peak location depends on the heating rate (Fig. 6). This effect is related to the Randall-Wilkins shift in thermoluminescence studies and can be used for the determination of activation energy and frequency factor of each trapping level. Because the accuracy of such calculations depends critically on the precision of the peak temperature determination, a sample (12033,46) has been measured in Braunschweig, Germany, in a differential precision reader designed by G. Holzapfel [6]. For a 1°C/sec. heating rate, the lowest peak was found to be at 130 ± 2°C, the second at 180 ± 3°C, and a high-temperature peak at 387 ± 3°C. These calibrations will be used for future calculations.

The thermal release of solar wind known to be trapped mainly in the surface regions (2000 Å) of the lunar surface fines has been used in a first attempt to explain the heating effect (80% of the hydrogen can be released by heating at 600°C). It was thought that it could perhaps occupy electron traps. Heating the lunar fines at 650°C, however, induces other permanent changes, as indicated by a color change, whose effects must be considered. In particular, reduced forms of iron are present initially which will be oxidized when heated. Weeks et al. [7] showed that iron ions in a variety of chemical and magnetic states are the sources of intense electron resonance absorption in lunar samples, and that partially reduced Fe_2O_3 gives a spectrum similar to that of the fines, breccias and glass spheres; there were changes in the resonance spectrum of the fines when heating at 600°C was conducted in air at 10^{-3} or 760 mm. Therefore, some similar experiments involving oxidation and reduction of the samples were performed on iron particles and Apollo 12 fines.

B. Oxidation and Reduction Studies

A portion of the lunar fines 12033,46 and analytical grade iron powder were heated at 650°C for 24 hours in a stream of 4% H_2, 96% Ar. As indicated in Fig. 7 a, both the fines and the iron powder initially did not exhibit any artificial TSEE. Repetitive heat cycles in the counter, however, developed an exoelectron response. For the fines, the principal change occurring in the glow curve structure was a shift of the peak at 200°C to higher temperatures to cause overlapping with the peak at 350°C. After the 6th heating cycle the two peaks had convolved to produce the single peak at 325°C. The reduced iron powder was oxidized at 650°C in air and upon γ-irradiation gave the TSEE curve shown in Fig. 7 c. The resemblance between the TSEE curves for the fines and the oxidized iron is obvious even if the absolute sensitivity of the iron powder is much below that of the lunar samples. One can infer from these results that some oxidation of the surface of both materials is necessary to develop TSEE capability. Even traces of oxygen in the counter (or adsorbed by the fines) seem to be sufficient to bring this about.

The trapped solar wind effect can now be reconsidered in light of the reduction experiments. During the heating in a 4% H_2 atmosphere at 650°C, much of the trapped solar wind will be released. Still there is no exoelectron activity, thus its presence initially in the surface regions of the fines cannot be the sole factor for the suppression of exoelectron activity. The buried H_2 may of course be the indirect cause, it having placed the surface regions in a preferentially reduced state. It appears that the lunar fines develop a TSEE capability when oxidized by heating in air, while this capability is suppressed when the heating is done in a hydrogen atmosphere.

Some parallel EPR measurements were made to study the corresponding bulk changes. The paramagnetic resonance spectra at 35 GHz are recorded in Fig. 8 and closely resemble those previously reported [7] for lunar fines, the resonance being attributed mainly to Fe^{3+}. Heating the fines 12033,46 causes a distinct broadening particularly on the low field side of the absorption peak. The spectra of the reduced and oxidized specimens, however, differ only in minor detail. The Fe^{3+} in the bulk of the particles remains largely untouched during the reduction treatment. It is, as expected, in the surface region responsible for TSEE activity that the reduction of iron ions occurs.

To corroborate the connection between TSEE and oxidation/reduction, the lunar fines 12033,46 were first oxidized to develop the TSEE activity and then reduced in H_2 to eliminate the exoelectron response. The result is shown in Fig. 9 as a decrease of the high temperature peaks; only minor changes in the TSEE curve can, however, be seen below 300°C: Some oxidation changes are reversible, others appear to be "locked into the solid". Clearly, the exoelectron response cannot be turned "on" and "off" by oxidation and reduction treatments. Referring back to Fig. 7, it can be seen that oxidation of the reduced fines 12033,46 produces an "unusual" looking glow curve especially with respect to the weak or absent low temperature peaks. Obviously, irreversible changes in the surface regions accompany the heat treatments.

In light of the sensitivity of the surface regions to oxidation processes, heating experiments were conducted in air to determine the temperature dependence of the TSEE sensitivity. Fines 12033,46 were heated at temperatures between 100°C and 500°C and the ensuing artificial

TSEE curves are shown in Fig. 10. Even at 100° a small amount of TSEE occurs. The surface temperature of the moon at the lunar noon is about 112°C. Heating at 100°C will not, therefore, be high enough to expel solar wind. This means that the induced TSEE arose through surface reactions with atmospheric constituents (oxidation). We have been concerned that degradation of the fines might be occurring in the atmosphere even at room temperature. Repeated checks have shown that, over a 4 month period, no artificial TSEE has developed.

Because the absence of TSEE in the fines could be due to buried solar wind producing a preferential state of reduction, material was sought which had not been exposed to solar wind. Accordingly, artificial TSEE curves have been measured for unheated particles scraped from an interior fracture surface of rock 12065,85. A TSEE curve (Fig. 11) was obtained which was very similar to the TSEE curves for the preheated and irradiated fines. This evidence appears to substantiate the hypotheses that solar wind in surface material has suppressed (indirectly) the TSEE response.

Severe localized frictional heating will undoubtedly have modified the nature of TSEE emitters in the surface produced by sawing of rock 12065,85. To study the effect further, particles were removed from one of the sawed faces and the artificial TSEE measured. The results are shown in Fig. 12. The first heating cycle in the counter shows that modifications have indeed occurred, particularly with respect to the relative population densities of trapping centers. With further heating, either in the counter or in air, the TSEE curve gradually reverts to one having the more normal shape. Annealing and oxidation processes could

account for the change.

C. Adsorption Effects

Heating (oxidation) in air at temperatures as low as 100°C produces TSEE capability in the lunar fines. Heating experiments were also conducted in vacuo (10^{-5} to 10^{-6} torr) to investigate whether the exoelectron capability was indeed dependent on an oxygen containing environment. This resulted in the discovery that another effect has importance for the development of artificial TSEE - adsorbed gases. The fines 12033,46 or 12070,18 were evacuated at 100°, 300° or 650°C. If after evacuation the fines were exposed to air and subsequently irradiated, weak TSEE signals could be recorded (Fig. 13). The strongest peak is the one at ~140°C. Apparently some change of the surface has taken place even during evacuation because irradiation of these same unheated fines produces no exoelectron response.

In contrast to the irradiation in air, irradiation of the outgassed fines in vacuo produced no TSEE signals. The two effects are shown in Fig. 14 for the fines 12070,18 evacuated at 300°C. One can conclude that adsorbed atmospheric species formed during irradiation are giving rise to at least part of the glow curve, and in particular the peak close to 150°C. A peak associated with an adsorbed species would be expected to vary in intensity with the surface area of the fines. Indeed if the fines are separated into coarse (lower area) and fine fractions (higher area), then the peak at ~150°C is more intense for the fine fraction. Also supporting the adsorbed oxygen theory are recent studies on ZnO[8], in which TSEE peaks in similar locations (127°C, 252°C, 457°C and above)

have been attributed, with high probability, to various adsorbed oxygen radicals.

In order to obtain more information on the surface area and structure, adsorption of N_2 was measured on the fines 12033,46. The specific surface area was <0.05 m^2/g corresponding to a mean particle size of >400 μm. A sample of fines with a higher area will be more suitable for adsorption studies related to TSEE, e.g. fines 10087,5 whose measured N_2 area is ~1,0 m^2 /g. The relatively coarse mean particle size of 12033,46 has been confirmed by some studies using a scanning electron-microscope (Fig. 15). Erosion and attachment of smaller particles seems to have enlarged the surface area of a small glassy sphere (Fig. 16). Some electrostatically attached small particles also became visible at higher magnifications on the surface of larger crystalline particles (Fig. 17).

A schematic summary of some of the factors affecting TSEE which have been studied so far is given in Figure 18.

Acknowledgments.

The authors gratefully acknowledge valuable experimental assistance and stimulating suggestions by numerous colleagues, in particular J. A. Auxier, H. F. Holmes, E. L. Fuller, Jr., R. K. Bennett, Jr., and R. A. Weeks, Oak Ridge; J. Kramer, G. Holzapfel and Miss R. Lange, Physikal.-Techn. Bundesanstalt, Braunschweig, Germany; and F. H. Attix and V. Ritz, U. S. Naval Research Laboratory, Washington, D. C.

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FIGURE CAPTIONS

Fig. 1. Effect of repetitive heating cycles (50 to 640°C) in counting gas (99.05% He, 0.95% isobutane) on radiation-induced TSEE in lunar surface fines 12033,46. The increasing emission above 600°C is due to thermionic electron emission.

Fig. 2. Effect of heating at 650°C in air on radiation-induced TSEE in surface fines 12070,18.

Fig. 3. Effect of heating at 650°C in air on TSEE response of core tube sample 12028,105 (parent 12028-32).

Fig. 4. Effect of repetitive heating in air and counting gas on total TSEE response between 75 and 600°C in two different surface fines.

Fig. 5. Total TSEE response of fines 12033, after a "stabilizing" pre-treatment for 30 min. at 640°C in counting gas, as a function of dose of ^{60}Co gamma radiation for all peaks between 75 and 600°C, for the low-temperature peaks (75 to 400°C), and for the high-temperature peaks (400 to 600°C).

Fig. 6. Heating-rate dependence of TSEE peak locations in fines 12033; peak temperatures uncorrected for gradient in graphite planchets.

Fig. 7. TSEE curves of lunar fines after reduction in H_2 during consecutive heating cycles in the counter (a and b), compared to the TSEE curve of reduced (a) and oxidized (c) iron powder.

Fig. 8. Effect of oxidation and reduction of the Electron Paramagnetic Resonance of fines 12033,46.

Fig. 9. Effect of oxidation for 1 hour at 600°C in air (a), followed by a reduction for 6 hours at 600°C in H_2/Ar, on the artificial (10^5 rad gamma radiation) TSEE in fines 12033,46.

Fig. 10. Effect of heating of virgin fines 12033,46 at various temperatures in air on their TSEE characteristics.

Fig. 11. TSEE response of unheated particles from the interior fracture surface of crystalline rock 12065,85 for an unirradiated sample, and for a different, but irradiated sample.

Fig. 12. Effect of different heat treatments on particles from the sawed faces of crystalline rock 12065,85.

Fig. 13. TSEE response of fines 12033,46 which were previously heated at 650°C in vacuo for 24 hours.

Fig. 14. TSEE response of fines 12070,18 heated in vacuo at 300°C for 24 hours followed by irradiation in vacuo or in air.

Fig. 15. Scanning Electron Micrograph (SEM) of crystalline particle (size ~0.3 mm) resembling the profile of a human head.

Fig. 16. SEM of glassy sphere (diameter ~0.5 mm) from surface fines.

Fig. 17. SEM of small particles attached to the surface of a larger crystalline fine particle.

Fig. 18. Schematical diagram of some major changes occurring in the TSEE characteristics of lunar materials.

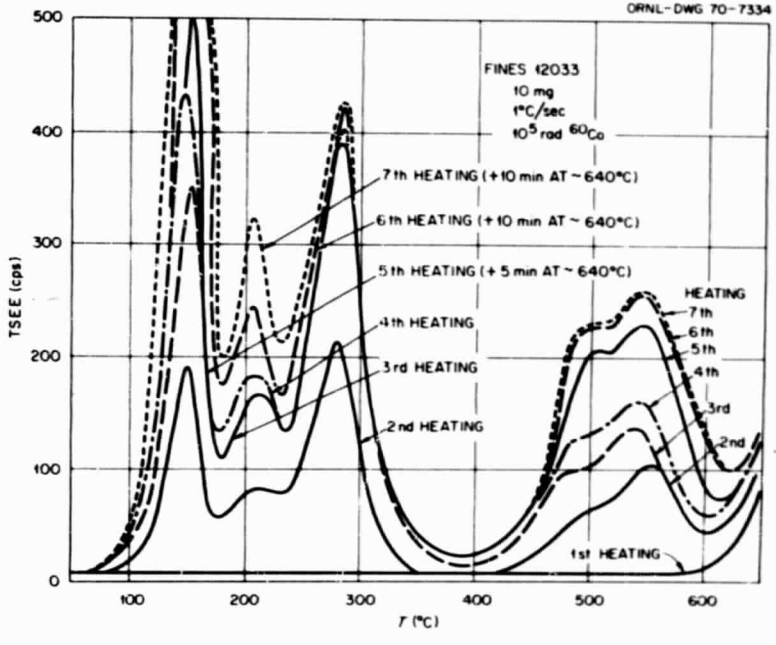

Fig. 1

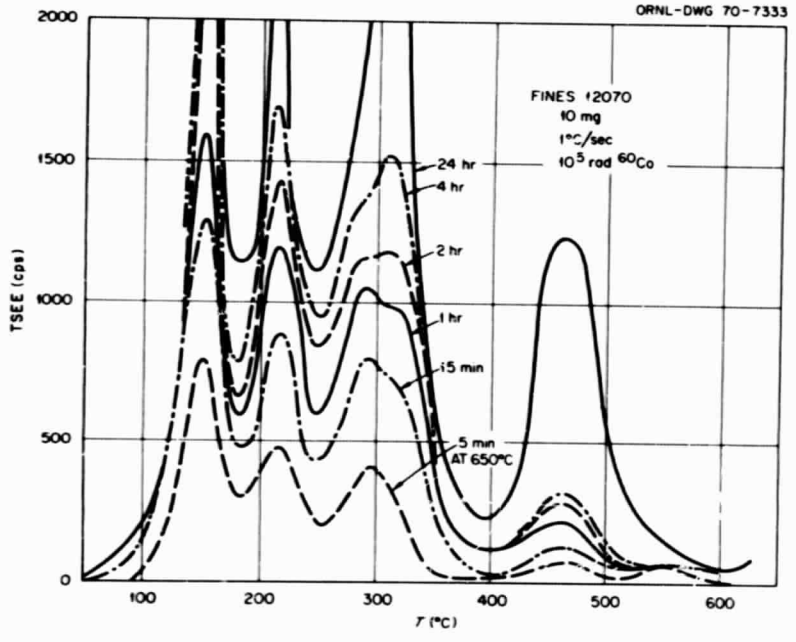
ORNL-DWG 70-7333
FINES 12070
10 mg
1°C/sec
10^5 rad 60Co
24 hr
4 hr
2 hr
1 hr
15 min
5 min AT 650°C
TSEE (cps)
T (°C)
2000
1500
1000
500
0
100
200
300
400
500
600

Fig. 2

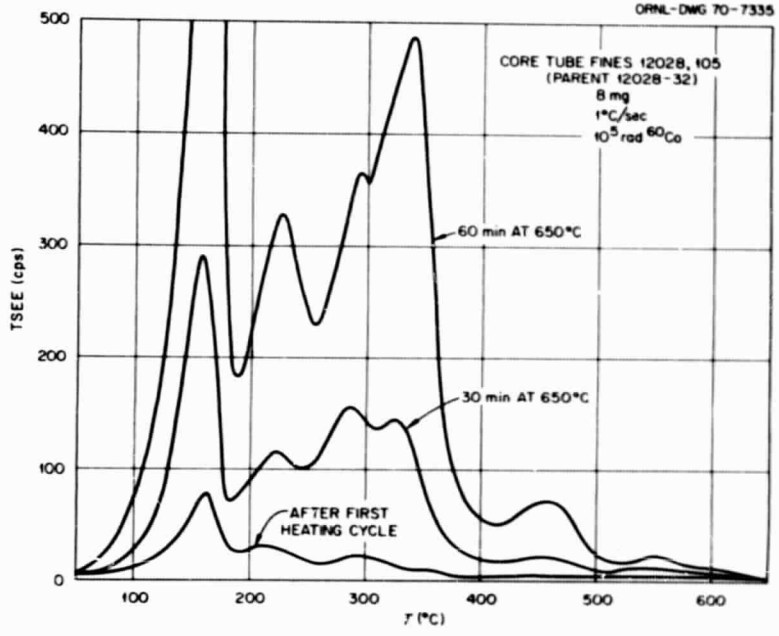
ORNL-DWG 70-7335
CORE TUBE FINES 12028, 105
(PARENT 12028-32)
8 mg
1°C/sec
10^5 rad 60Co
60 min AT 650°C
30 min AT 650°C
AFTER FIRST
HEATING CYCLE
TSEE (cps)
T (°C)
500
400
300
200
100
0
100
200
300
400
500
600

Fig. 3

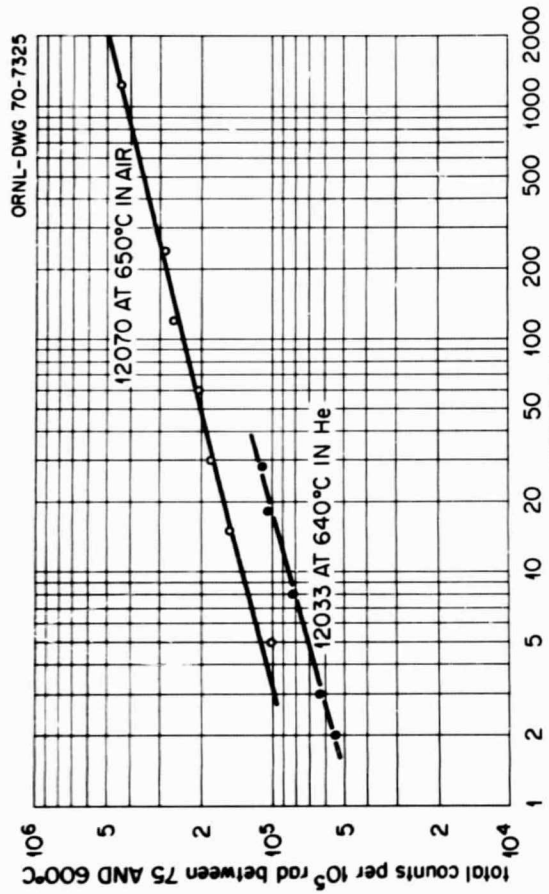
ORNL-DWG 70-7325
12070 AT 650°C IN AIR
12033 AT 640°C IN He
TIME (min)
1
2
5
10
20
50
100
200
500
1000
2000
total counts per 10^5 rad between 75 AND 600°C
10^6
5
2
10^5
5
2
10^4

Fig. 4

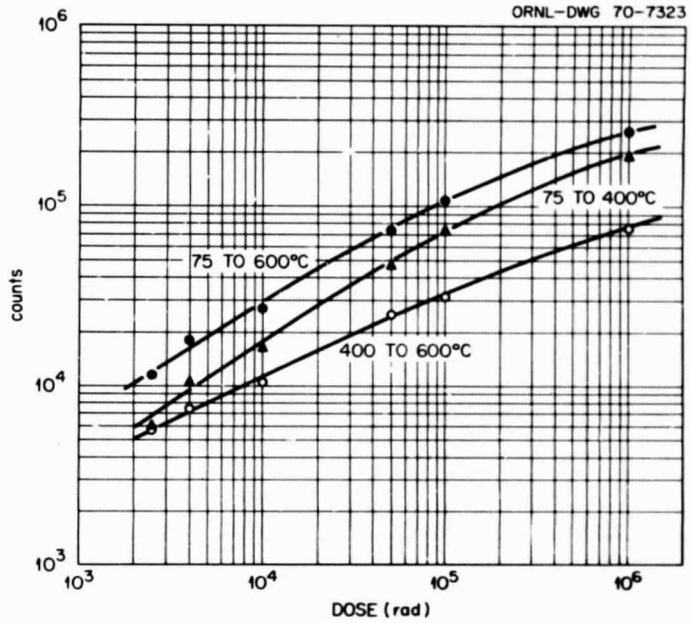
ORNL-DWG 70-7323
10^6
10^5
10^4
10^3
counts
75 TO 600°C
75 TO 400°C
400 TO 600°C
10^3
10^4
10^5
10^6
DOSE (rad)

Fig. 5

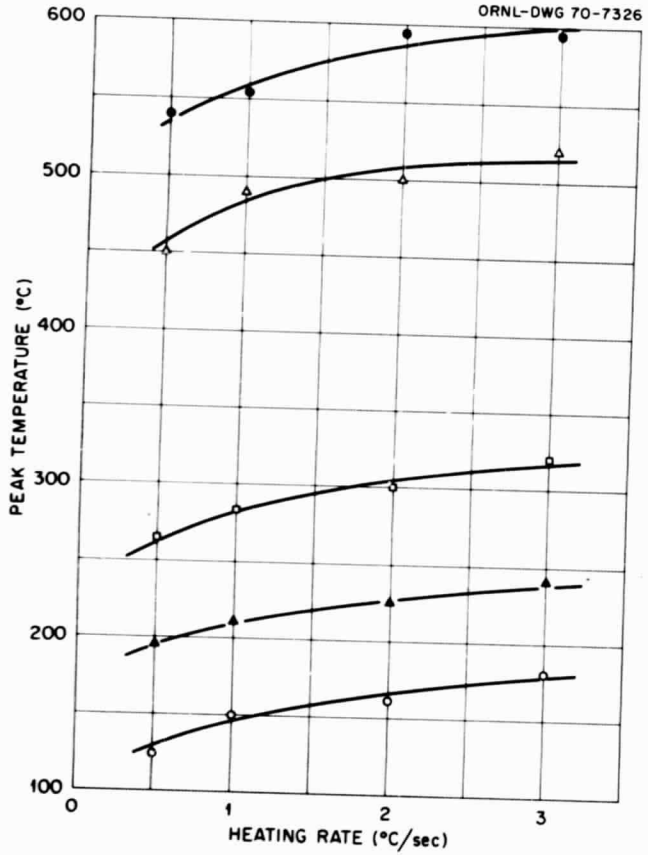
ORNL-DWG 70-7326
PEAK TEMPERATURE (°C)
HEATING RATE (°C/sec)
600
500
400
300
200
100
0
1
2
3

Fig. 6

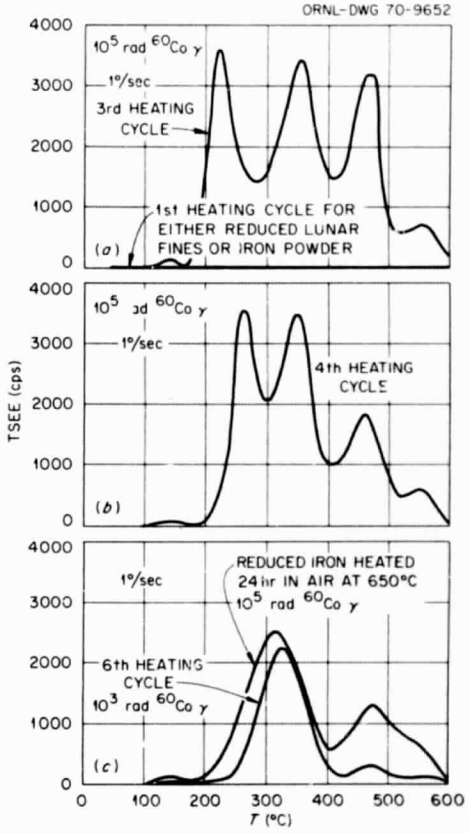
ORNL-DWG 70-9652
TSEE (cps)
T (°C)
10⁵ rad ⁶⁰Co γ
1°/sec
3rd HEATING CYCLE
1st HEATING CYCLE FOR EITHER REDUCED LUNAR FINES OR IRON POWDER
(a)
10⁵ rad ⁶⁰Co γ
1°/sec
4th HEATING CYCLE
(b)
1°/sec
REDUCED IRON HEATED 24 hr IN AIR AT 650°C
10⁵ rad ⁶⁰Co γ
6th HEATING CYCLE
10³ rad ⁶⁰Co γ
(c)
4000
3000
2000
1000
0
100
200
300
400
500
600

Fig. 7

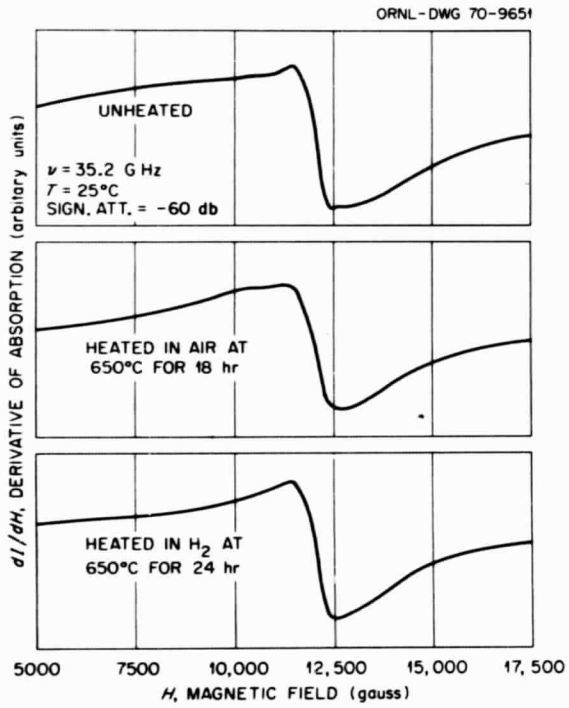
ORNL-DWG 70-9651
UNHEATED
ν = 35.2 G Hz
T = 25°C
SIGN. ATT. = -60 db
HEATED IN AIR AT
650°C FOR 18 hr
HEATED IN H2 AT
650°C FOR 24 hr
dI/dH, DERIVATIVE OF ABSORPTION (arbitary units)
5000
7500
10,000
12,500
15,000
17,500
H, MAGNETIC FIELD (gauss)

Fig. 8

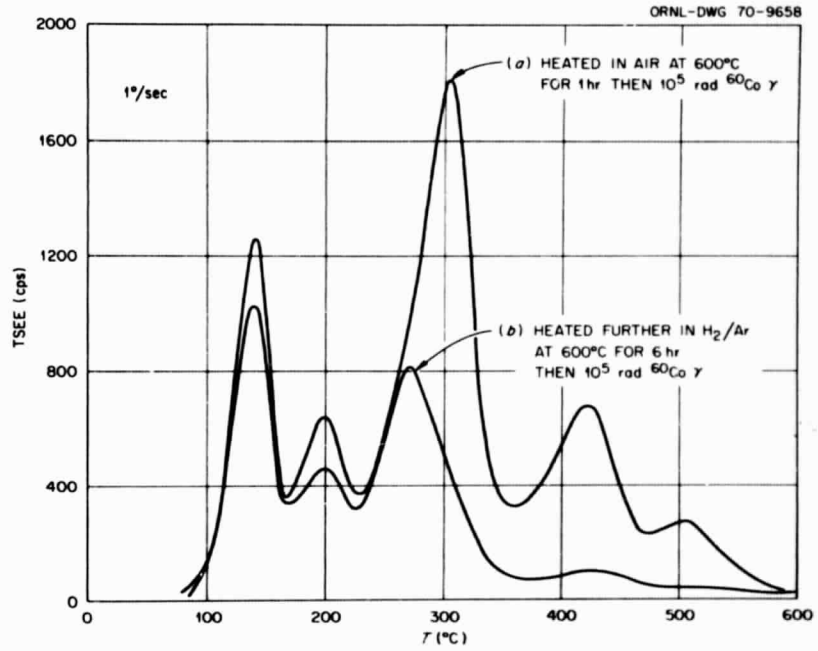
ORNL-DWG 70-9658
1°/sec
(a) HEATED IN AIR AT 600°C
FOR 1 hr THEN 10^5 rad 60Co γ
(b) HEATED FURTHER IN H2/Ar
AT 600°C FOR 6 hr
THEN 10^5 rad 60Co γ
TSEE (cps)
2000
1600
1200
800
400
0
0
100
200
300
400
500
600
T (°C)

Fig. 9

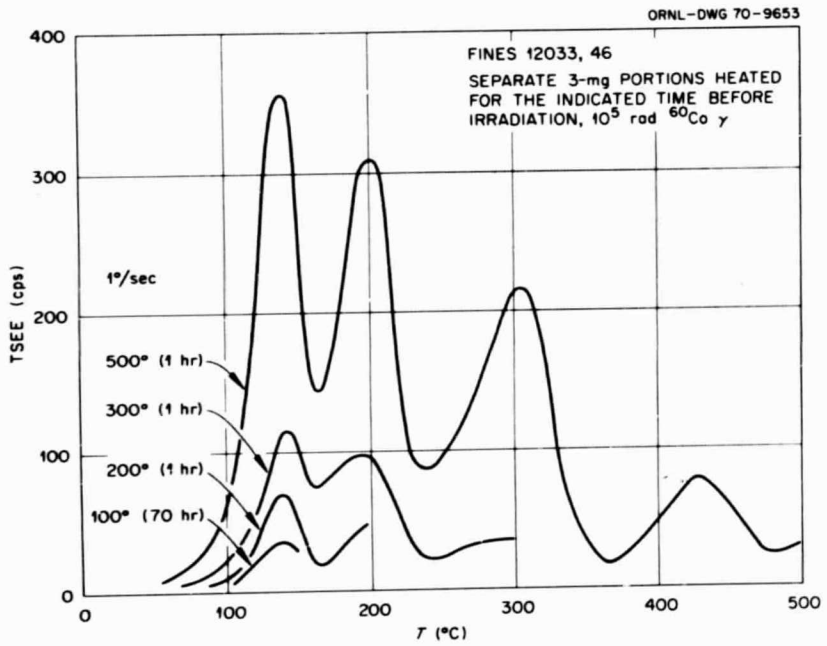
ORNL-DWG 70-9653
FINES 12033, 46
SEPARATE 3-mg PORTIONS HEATED FOR THE INDICATED TIME BEFORE IRRADIATION, 10^5 rad ^{60}Co γ
1°/sec
500° (1 hr)
300° (1 hr)
200° (1 hr)
100° (70 hr)
TSEE (cps)
T (°C)
0
100
200
300
400
500

Fig. 10

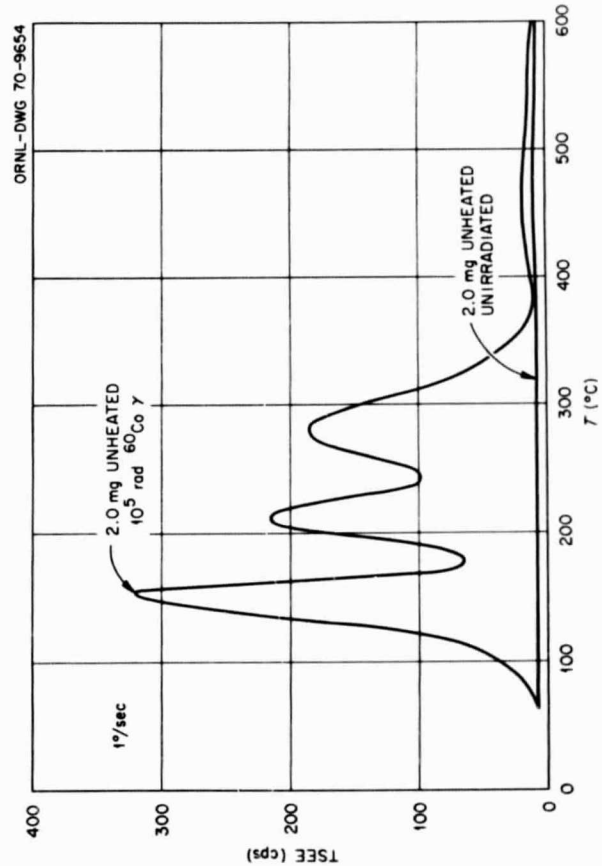
ORNL-DWG 70-9654
2.0 mg UNHEATED UNIRRADIATED
2.0 mg UNHEATED 10^5 rad ^{60}Co γ
1°/sec
TSEE (cps)
T (°C)
0
100
200
300
400
500
600

Fig. 11

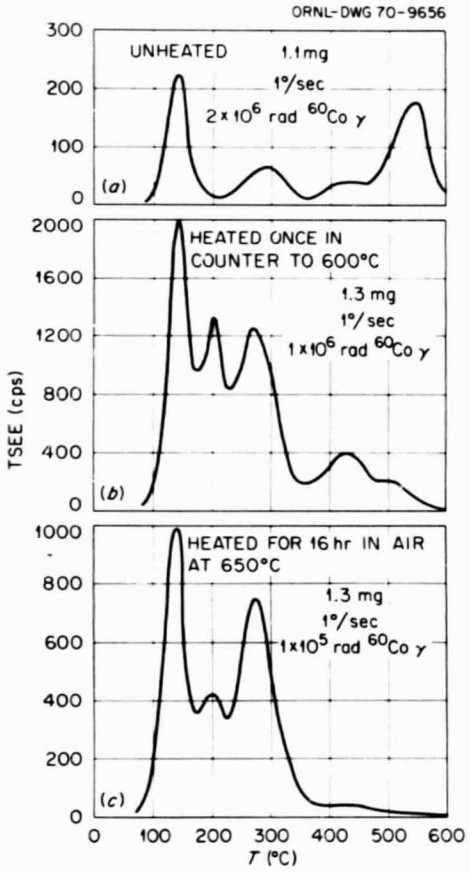
ORNL-DWG 70-9656
UNHEATED
1.1 mg
1°/sec
2 x 10^6 rad 60Co γ
(a)
HEATED ONCE IN COUNTER TO 600°C
1.3 mg
1°/sec
1 x 10^6 rad 60Co γ
(b)
HEATED FOR 16 hr IN AIR AT 650°C
1.3 mg
1°/sec
1 x 10^5 rad 60Co γ
(c)
TSEE (cps)
T (°C)

Fig. 12

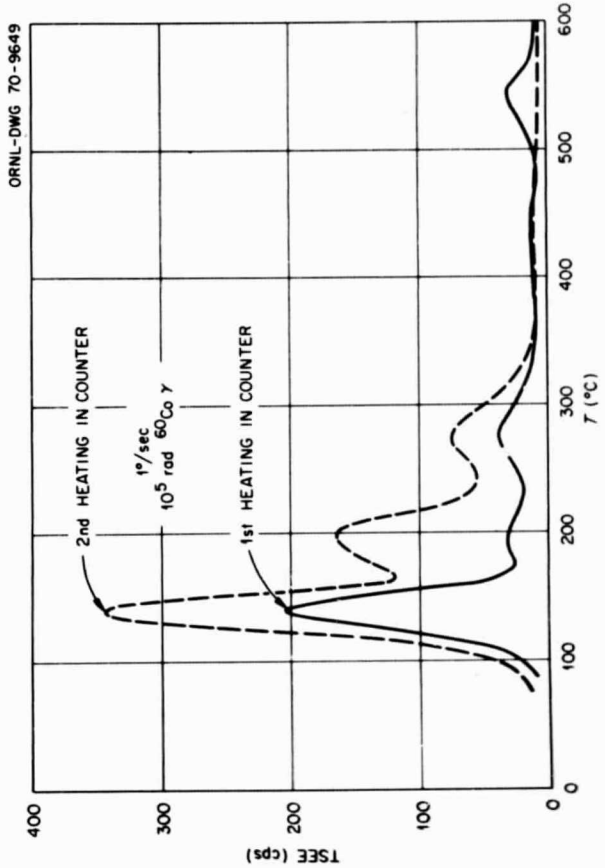
ORNL-DWG 70-9649
2nd HEATING IN COUNTER
1°/sec
10^5 rad 60Co γ
1st HEATING IN COUNTER
TSEE (cps)
T (°C)

Fig. 13

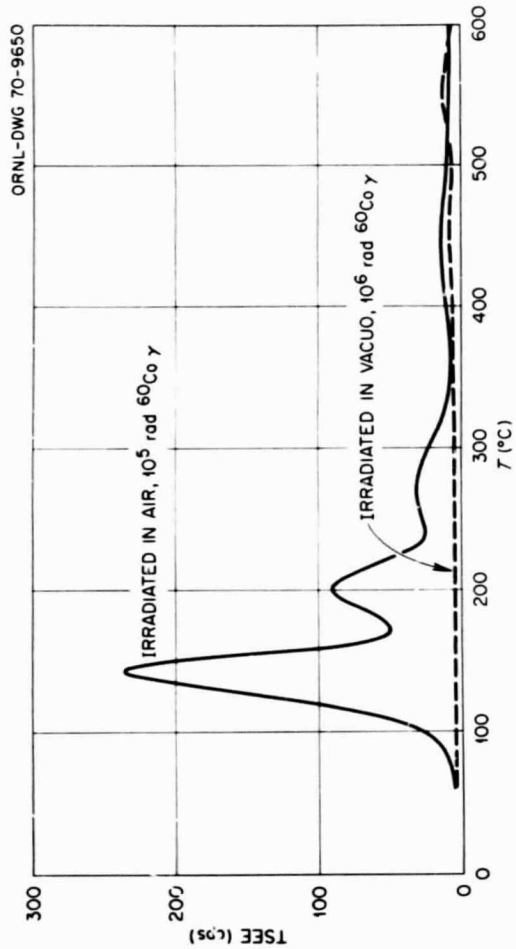
ORNL-DWG 70-9650
IRRADIATED IN AIR, 10^5 rad ^{60}Co γ
IRRADIATED IN VACUO, 10^6 rad ^{60}Co γ
TSEE (cps)
T (°C)
0
100
200
300
400
500
600

Fig. 14

PHOTO 135477

Fig. 15

Fig. 16

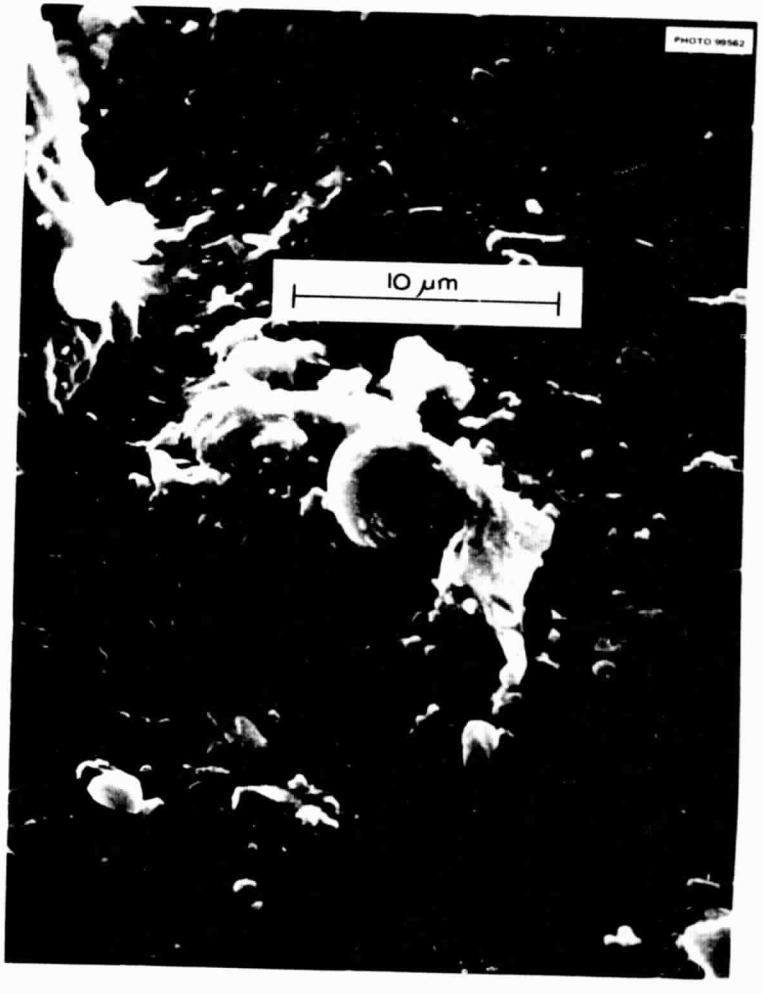

Fig. 17

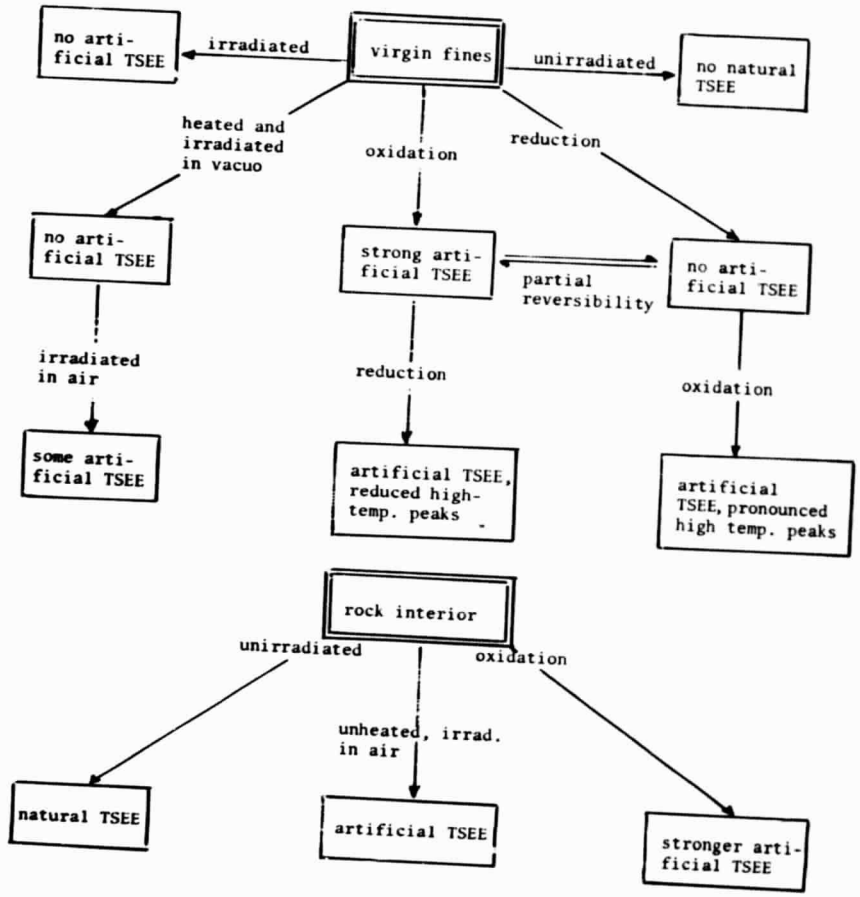
virgin fines
irradiated
no artificial TSEE
unirradiated
no natural TSEE
heated and irradiated in vacuo
oxidation
reduction
no artificial TSEE
strong artificial TSEE
partial reversibility
no artificial TSEE
irradiated in air
reduction
oxidation
some artificial TSEE
artificial TSEE, reduced high-temp. peaks
artificial TSEE, pronounced high temp. peaks
rock interior
unirradiated
oxidation
unheated, irrad. in air
natural TSEE
artificial TSEE
stronger artificial TSEE

Fig. 18