DETERMINING THE PHYSICAL PROPERTIES OF STEAM GENERATOR TUBE SCALE USING MINIATURE SPECIMENS

MATERIALS

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Small flakes that consist primarily of magnetite have been discovered on the secondary side of the steam generator of the Three Mile Island Unit 1 plant. These iron oxide flakes are believed to cause significant increases in flow resistance, which in turn causes abnormal increases in steam generator water level. It is necessary to measure the physical properties of the tube scale so that the maximum amount of loose flakes can be generated prior to hydrodynamic cleaning (water slap). It is also important to study the flake properties to shed light on the flake formation and transport mechanisms. Once the physical properties of the tube scale are determined, the effects of hydrodynamic cleaning (water slap) can be optimized by preconditioning the scale. There are several preconditioning options including prewetting, predrying, and thermal cycling of the steam generator tubes. Understanding the physical properties of the scale would also be beneficial in optimizing the water slap technique itself.

Elastic modulus, fracture stress, thermal expansion, and swelling of the flakes were measured. With one exception, all of the flakes studied were either one- or two-layered as judged by microstructural variation. The fracture stress of the flake materials tested was in the range of 20.0 to 113.8 MPa (2.9 to 16.5 ksi). There did not appear to be a substantial change in the range of stresses measured at elevated temperatures. There was no evidence of delamination during bend testing. The mean coefficient of linear thermal expansion was a factor of ~2 larger than that of Fe_3O_4. The maximum amount of swelling measured was 0.0012%, which is consistent with earlier data on flakes from the Oconee-2 plant.

INTRODUCTION

Small iron oxide (magnetite) flakes similar to those found in the steam generator at the Oconee-2 plant were found at General Public Utilities' (GPU's) Three Mile Island Unit 1 (TMI-1) plant. These flakes are believed to cause significant increases in flow resistance, which in turn causes abnormal increases in the steam generator water level. The effects of oxide spalling are discussed in Ref. 1, and more recent information is provided in Ref. 2. Measurements of the physical properties of this tube scale were made to amplify the results of the Oconee-2 measurements[3,4] and provide data necessary for optimization of the TMI-1 steam generator cleaning process. Using Battelle's patented miniature specimen technique,[5] elastic modulus, fracture stress, thermal expansion, and swelling of the TMI-1 flakes were measured.[a]

SPECIMEN PREPARATION

A metallographic study was conducted to investigate the microstructure of the flakes and to determine whether the flakes are multilayered. Sludge from the steam generator was sorted and 11 large flakes were placed in vials marked G1 through G11, a designation used throughout the program. The flakes were selected at random and are believed to be representative of the

[a]The techniques described are explained in part in U.S. Patent No. 4,567,774 dated February 4, 1986.

entire lot of flakes. A small chip cut from each of the flakes was metallographically mounted, polished, and etched. The etchant that best revealed the grain structure was 45% H_2O, 30% HCl, 20% HNO_3, and 5% HF. All the specimens investigated had at most two layers, with one exception. The criterion used to judge the number of layers present in the specimens was an abrupt change in the average grain size.[3] Table I presents the results of the metallography investigation, and Figs. 1, 2, and 3 show representative photomicrographs of the flake specimens. Figure 2 shows a two-layered specimen that is evident from the large-diameter grains near the inner diameter and smaller grains near the outer diameter surface. All of the specimens examined after polishing had particles (possibly copper-rich) as shown in Fig. 3 of Ref. 3. Evidence of a possible glassy phase was also observed during the light microcscopy investigation. Specimen G9 appeared to have a different material near the center of the cross section. The material was removed after etching.

The specimens were machined as described in Ref. 3. The bend specimens were machined so that their length dimension was parallel to the hoop direction of the Inconel tubes, while the swelling and thermal expansion specimens were oriented so that their length was parallel to the tube axis.

Since the TMI-1 flakes were much thinner than the Oconee-2 flakes, a higher percentage were fractured

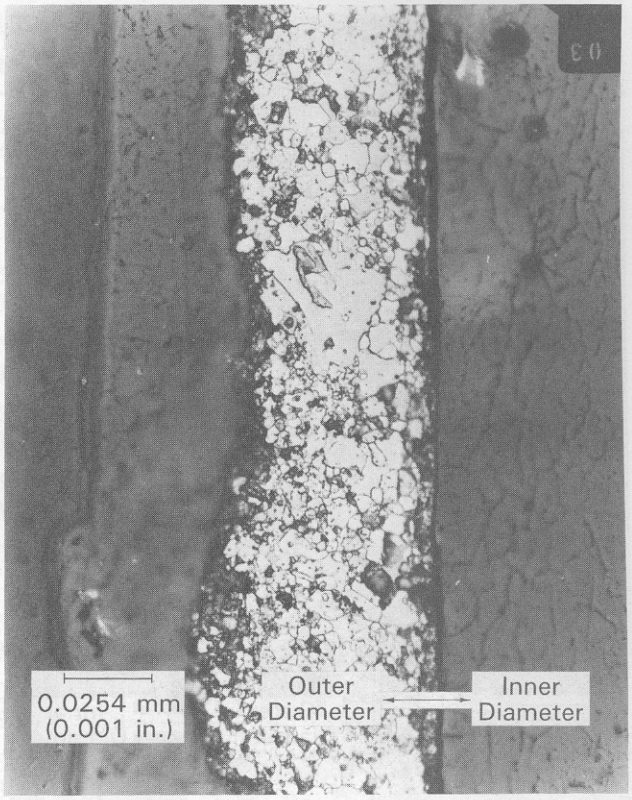

Fig. 1. Photomicrograph of specimen G3 showing single layer of large grains intermixed with small grains.

TABLE I

Results of Metallography Investigation

Specimen Identification	Number of Layers	Layer Characteristics[a]		Physical Property Test
		Region 1[b,c]	Region 2	
G1	2	SG ($\frac{1}{4}T$)	LG	Thermal expansion, swelling
G2	2	SG ($\frac{1}{5}T$)	LG	Thermal expansion
G3	1	LGSG	---	Mechanical
G4	2	MG	LGPL	Mechanical
G5	1	LGSG	---	Mechanical
G6	2	SG ($<\frac{1}{4}T$)	LG	Mechanical
G7	2	SG ($<\frac{1}{4}T$)	LGSG	Thermal expansion, swelling
G8	1	LGSG	---	Mechanical
G9	central region	SG (inner diameter)	LG (outer diameter)	Thermal expansion, swelling
G10	1	LGSG	---	Thermal expansion, swelling
G11	1	LGSG	---	Mechanical

[a]Here, LGPL = large-grained porous layer
LG = large grains
SG = small grains
LGSG = large grains intermixed with small grains
MG = medium grains.

[b]For two-layered specimens, the small-grain region is the inner diameter surface.

[c]T = thickness of the specimen.

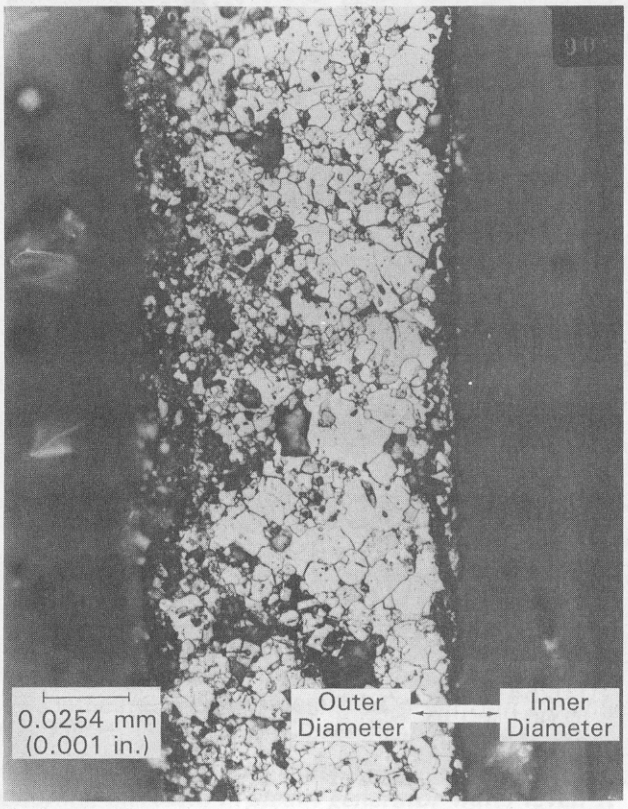

Fig. 2. Photomicrograph of specimen G6 showing two layers and increasing porosity near the outer diameter surface.

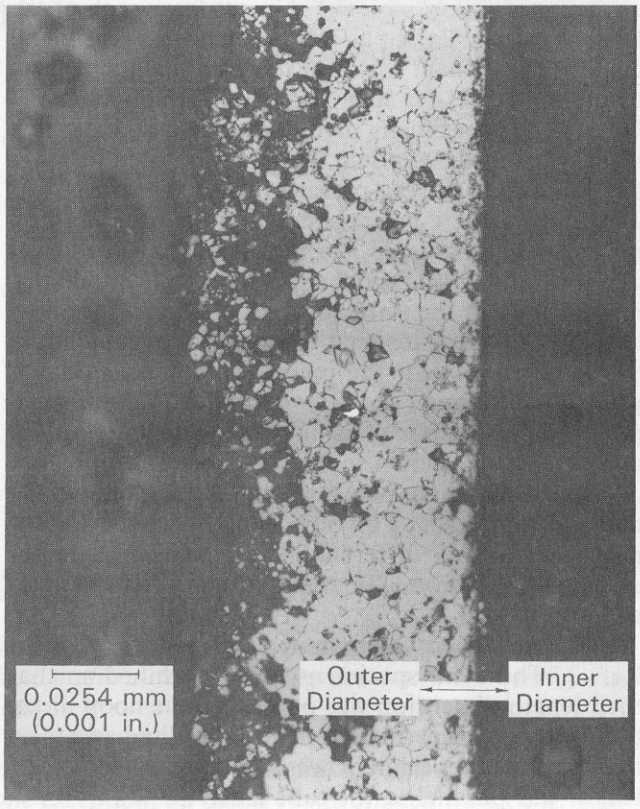

Fig. 3. Photomicrograph of specimen G7 showing a particle that remained after etching.

during handling. A second batch of flakes was sent to Battelle and additional mechanical behavior specimens were machined. These specimens were placed in vials marked G50-1 through G50-7, designations used throughout the program. The dimensions of each specimen are given in Table II.

METHODS AND RESULTS

Elastic Modulus and Fracture Stress

A static bend test was used to measure fracture strength and elastic modulus. For a brittle material such as magnetite, four-point bend loading is preferable to three-point loading since a brittle specimen in a three-point bend test may not fracture at the point of maximum bending moment (i.e., near the point of load application). However, in a four-point bend test, the entire region between the two loading points is at a constant maximum value of bending moment. Provided the test specimen fractures between the two load points, a constant value of maximum bending moment may be used in the continuum equations for fracture strength and modulus.

The apparatus and test procedures used are described in Ref. 3. Tests were conducted at room temperature (RT) [20°C (68°F)] and at 288°C (550°F). As discussed in Ref. 3, the difference between flat plate and curved beam solutions is small (~1%), and, therefore, flat plate equations were used to analyze the data. In all tests, the load train compliance was measured [RT and 288°C (550°F)], and the data were adjusted for compliance. The specimen preload (deadweight of the punch) was added to the recorded data. The measured punch, support, and specimen dimensions after machining were used in the calculations. The load cell and linear variable-differential transducer were calibrated to maximize sensitivity and accuracy. The estimated uncertainty in load measurement is $\pm 10.0 \times 10^{-4}$ kg ($\pm 2.2 \times 10^{-3}$ lb) for a calibration range of 0 to 0.40 kg (0 to 0.88 lb). The estimated uncertainty in deflection measurement is $\pm 13 \times 10^{-5}$ mm ($\pm 5.0 \times 10^{-6}$ in.) for a calibrated range of 0.03 mm (1.0×10^{-3} in.). The measurements were stored on computer for analysis and plotting.

Data from bend tests on large porcelain specimens

TABLE II

Miniature Specimen Dimensions at Room Temperature, 20°C (68°F)

Specimen Identification	Specimen Type[a]	Length [mm (in.)]	Width [mm (in.)]	Thickness [mm (in.)]
G1-1	S	---	---	0.0470 (0.00185)
G3-1	MB	2.1252 (0.08367)	1.9566 (0.07703)	0.0714 (0.00281)
G4-1	MB	2.1252 (0.08367)	1.8357 (0.07227)	0.0975 (0.00384)
G4-3	MB	2.1252 (0.08367)	1.5603 (0.06143)	0.0958 (0.00377)
G5-1	MB	2.1252 (0.08367)	1.9121 (0.07528)	0.0881 (0.00347)
G6-1	MB	3.070352 (0.12088)	1.9497 (0.07676)	0.0922 (0.00363)
G6-2	MB	2.1252 (0.08367)	1.8961 (0.07465)	0.0970 (0.00382)
G7-1	TE,S	7.57072 (0.29806)	---	0.0930 (0.00366)
G9-1	TE,S	3.8964 (0.15340)	---	0.0805 (0.00317)
G10-1	TE,S	2.77419 (0.10922)	---	0.0711 (0.00280)
G50-1	MB	2.1252 (0.08367)	1.3579 (0.05346)	0.109 (0.00427)
G50-2	MB	2.3292 (0.09170)	1.4465 (0.05695)	0.0780 (0.00307)
G50-3	MB	1.4272 (0.05619)	1.6228 (0.06389)	0.0899 (0.00354)
G50-4	MB	1.7620 (0.06937)	1.8227 (0.07176)	0.0767 (0.00302)
G50-6	MB	1.7488 (0.06885)	1.4249 (0.05610)	0.0886 (0.00349)
G50-7	MB	1.5540 (0.06118)	1.5326 (0.06034)	0.0993 (0.00391)

[a]Here, MB = mechanical behavior
TE = thermal expansion
S = swelling.

were compared with the miniature specimen data (Table III). The average miniature bend modulus was ~55% lower than the average large specimen modulus determined in compression. This difference is probably due to the measurement technique (bending versus compression) and the uncertainty in the specimen thickness and surface condition in the vicinity of the crack. The average miniature specimen fracture stress was ~15% lower than that for the large specimens. However, the miniature specimen fracture stress data fall within the 1-σ uncertainty band. The lower value for the miniature specimen fracture stress reported in Ref. 3 for porcelain is probably due to specimen thickness uncertainties and also the effects of surface scratches. The specimens in the current study had a better surface finish than those of Ref. 3. Although the specimens were polished, small scratches in the miniature specimens are likely to have a greater

TABLE III

Porcelain Benchmark Data for Miniature Four-Point Bend Test

Specimen Identification	Miniature Specimens		Large Specimens	
	Maximum Stress [MPa (psi)]	Elastic Modulus, Bending [MPa ($\times 10^6$ psi)]	Maximum Stress [MPa (psi)]	Elastic Modulus [MPa ($\times 10^6$ psi)]
P1	67 (9712)	35779 (5.19)	---	---
P2	83 (12086)	50739 (7.36)	---	---
P3	50 (7278)	33022 4.79	---	---
P4	55 (8047)	34125 4.95	---	---
P5	50 (7188)	26886 3.90	---	---
P6	73 (10563)	31229 4.53	---	---
P8	51 (7430)	34125 4.95	---	---
P9	66 (9624)	29644 4.30	---	---
Average	62 (8991)	34444 5.00	72 (10514) (bending)	71008 (10.3) (uniaxial compression) 73076 (10.6) (pulse/echo)
Standard deviation	11.5 (1676)	7172 (1.04)		
Average from Ref. 3	47 (6770)	48947 (7.10)		

effect on the fracture stress than similar scratches in large specimens.

The miniature flake data are presented in Tables IV and V for the RT and 288°C (550°F) tests, respectively. As mentioned in Ref. 1, conclusive statements concerning trends in the data cannot be made at present. Further research is required to quantify some of the behavior that was observed during testing. Overall, the flake response under loading appears to be complex. The effects observed during testing and some plausible explanations for them are provided below. These plausible explanations, however, have not been completely confirmed by experiment.

The actual thickness at the fracture surface was measured after the test and used in the calculations. In the Oconee-2 flake study,[3,4] some of the flakes delaminated during the test, and the layer thicknesses were used to correct the data. There was no evidence of delamination in the current study. Also, a porous layer correction was applied to the data for the Oconee-2 flakes. Since the width of the outer layer varied significantly and the outside diameter layer of the TMI flakes was not as porous as the Oconee-2 flakes, no porous layer correction was applied to the TMI data.

It is not possible to report an elastic modulus in

TABLE IV

Mechanical Behavior of Flakes at Room Temperature 20°C (68°F)

Specimen Identification	Orientation	Specimen Thickness [mm (in. × 10^{-3})]	Maximum Stress [MPa (psi)]	Flake Modulus [MPa (× 10^6 psi)]
G41U	Concave up	0.0975 (3.84)	50 (7 247)	38 330 (5.56)
G43U	Concave up	0.0958 (3.77)	47 (6 759)	42 398 (6.15)
G51U	Concave up	0.0881 (3.47)	77 (11 124)	25 783 (3.74)
G61D	Concave down	0.0922 (3.63)	114 (16 500)	99 963 (14.50)
G503D	Concave down	0.0899 (3.54)	21 (2 992)	72 593 (10.53)
G504D	Concave down	0.0767 (3.02)	42 (6 117)	30 057 (4.36)
Average		0.0900 (3.55)	59 (8 558)	51 521 (7.47)
Standard deviation		0.0074 (0.29)	33 (4 725)	28 883 (4.20)

TABLE V

Mechanical Behavior of Flakes at 288°C (550°F)

Specimen Identification	Orientation	Specimen Thickness [mm (in. × 10^{-3})]	Maximum Stress [MPa (psi)]	Flake Modulus [MPa (× 10^6 psi)]
G31U	Concave up	0.0714 (2.81)	42 (6 102)	43 225 (6.27)
G501U	Concave up	0.108 (4.27)	36 (5 164)	13 305 (1.93)
G62U	Concave up	0.0970 (3.82)	77 (11 123)	47 775 (6.93)
G502D	Concave down	0.0780 (3.07)	199[a] (28 840)	367 588[a] (53.32)
G506D	Concave down	0.0886 (3.49)	84 (12 211)	330 567[b] (47.95)
G507D	Concave down	0.0993 (3.91)	79 (11 480)	113 613 (16.48)
Average		0.0904 (3.56)	64 (9 225)	54 480 (7.90)
Standard deviation		0.0138 (0.54)	23 (3 293)	42 284 (6.13)

[a]Data may not be valid due to probable fracture outside of punch span; average does not include these data.
[b]Modulus data are not valid due to irregular crack propagation; average does not include this datum.

the conventional sense. The elastic modulus is a measure of the displacement of atoms from their equilibrium lattice positions in a given material. Since a given flake may have compositional variations through the thickness and varying grain sizes (indicating different mechanical response within each layer), the modulus was calculated based on the slope of the load deflection curve and is called the "flake modulus." Large variations in the flake modulus are to be expected.

The maximum stress at room temperature of the TMI-1 specimens ranged from ~21 to 114 MPa (3000 to 16 500 psi), and there was no discernible difference between tests conducted concave up or down. The Oconee-2 data were somewhat higher and ranged from ~52 to 162 MPa (7500 to 23 500 psi). The maximum stress at 288°C (550°F) ranged from ~34 to 83 MPa (5000 to 12 000 psi), as compared with the Oconee-2 data, which ranged from ~51 to 117 MPa (7500 to 17 000 psi). The scatter in the data is large, as shown in Tables IV and V. This is due to the fact that the flakes were tested in the as-received condition. Variations in the porosity and surface condition have a significant effect on mechanical response.

Thermal Expansion

Thermal expansion was measured optically using a microscope with a precision x-y stage attached to a digital micrometer. The system was designed so that backlash is essentially zero and the stage was calibrated so that the measurement uncertainty is much lower than the uncertainty in cross hair placement. The heating apparatus and measurement procedure used were the same as reported in Ref. 4.

The linear thermal expansion data for the TMI-1 flakes are presented in Table VI, and the data are compared with baseline data for Fe_3O_4 (Ref. 6) in Fig. 4. The mean coefficient of linear expansion over several temperature ranges is provided in Table VII. The instantaneous coefficient of linear expansion at several temperatures is provided in Table VIII. During the testing, nine readings were taken for length measurements at each temperature and the readings averaged. The average standard deviation for the deflection data was consistently ~0.00290 mm (~1.14×10^{-4} in.).

The higher temperature data are in error due to specimen curling.[3] Specimen curling was shown to be negligible for the Oconee-2 flakes up to ~204°C (399°F). Curling data would be necessary to confirm this finding for the TMI-1 specimens. Since these specimens are substantially thinner than the Oconee-2 flakes, curling may be more significant. The data taken at 316°C (601°F) were not valid due to curling.

Consistent with the data reported in Refs. 3 and 4, the flake expansion coefficients are higher than those of Fe_3O_4. The single-layered specimen appears to have a larger expansion coefficient than the two-layered specimen; however, additional data are needed to confirm this possible trend. The data reported here are likely to be lower bound estimates since a curling correction was not applied.

TABLE VI

Linear Thermal Expansion Data for TMI-1 Miniature Flake Specimens

Specimen Identification	Number of Layers	Thickness [mm (in. × 10^{-3})]	$\Delta L/L_0$ (%)[a]				
			RT to 93°C (RT to 200°F)	RT to 149°C (RT to 300°F)	RT to 204°C (RT to 400°F)	RT to 260°C (RT to 500°F)	RT to 316°C (RT to 600°F)
G71	2	0.0930 (3.66)	0.16440	0.22143	0.27176	0.33550	---
G91	2[b]	0.0805 (3.17)	0.04563	0.24772	0.33246	0.31943	0.05215[c]
G101	1	0.0711 (2.80)	0.20143	0.31130	0.46695	0.60428	0.25636[c]
Average for flakes G71, G91, and G101	---	---	0.13715	0.26015	0.35706	0.41974	---
Ref. 4 data for Fe_3O_4	---	---	0.067	0.121	0.182	0.251	0.325

[a]The term L_0 is referenced to RT, 21°C (70°F).
[b]This specimen had a central region that could be a glassy phase.
[c]Evidence of specimen curling.

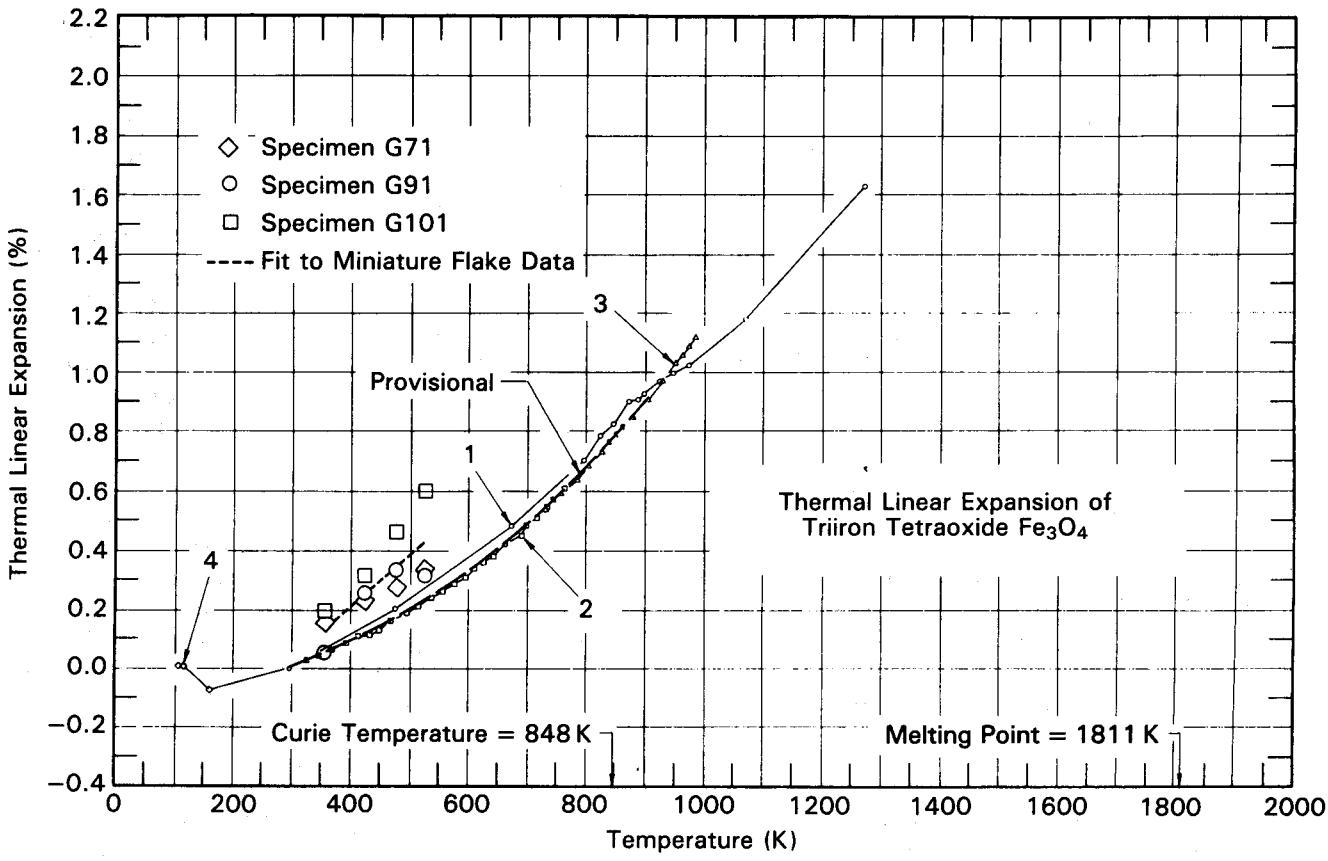

Fig. 4. Comparison of TMI-1 miniature flake thermal expansion data with Fe_3O_4 data.[6]

TABLE VII

Mean Coefficient of Linear Thermal Expansion for TMI-1 Miniature Flake Specimens

Specimen Identification	Mean Expansion Coefficient, α [$°C^{-1} \times 10^{-5}$ ($°F^{-1} \times 10^{-6}$)]				
	RT to 93°C (RT to 200°F)	RT to 149°C (RT to 300°F)	RT to 204°C (RT to 400°F)	RT to 260°C (RT to 500°F)	RT to 316°C (RT to 600°F)
Average for flakes G71, G91, and G101	1.91 (10.55)	2.03 (11.31)	1.95 (10.82)	1.76 (9.76)	---
Ref. 4 data for Fe_3O_4	0.92 (5.08)	0.94 (5.22)	0.99 (5.48)	1.05 (5.81)	1.10 (6.11)

Swelling

The swelling measurement procedure described in Ref. 3 was modified to improve accuracy. A Sheffield Accutron (model 50185) vertical comparator was used and all measurements were made in a temperature- and humidity-controlled laboratory. Calibration runs were made prior to taking measurements on a specimen. The stylus was brought into contact with a steel specimen blank that was approximately the same length as the flake specimens and held until equilibrium was established. Water was added to the stage and readings taken every half hour to obtain the calibration curve. The specimen, a portion of the stylus, and the water pool were sealed with cellophane to provide an equilibrium vapor pressure. This resulted in much less shortening of the stylus due to evaporation.

The water was then removed and the specimen

TABLE VIII

Instantaneous Coefficient of Linear Thermal Expansion for TMI-1 Miniature Flake Specimens

Specimen Identification	Expansion Coefficient, α [$°C^{-1} \times 10^{-5}$ ($°F^{-1} \times 10^{-6}$)]							
	57°C (135°F)	121°C (250°F)	149°C (300°F)	177°C (350°F)	204°C (400°F)	232°C (450°F)	260°C (500°F)	343°C (650°F)
Average for flakes G71, G91, and G101	1.91 (10.55)	2.2 (12.30)	1.98 (11.00)	1.76 (9.69)	1.44 (7.98)	1.12 (6.27)	---	---
Ref. 4 data for Fe_3O_4	0.92 (5.08)	0.96 (5.40)	1.04 (5.75)	1.11 (6.10)	1.17 (6.50)	1.23 (6.90)	1.28 (7.15)	1.32 (7.40)

TABLE IX

Swelling Data for TMI-1 Flakes

Time (h)	Specimen Elongation Data [mm $\times 10^{-5}$ (in. $\times 10^{-6}$)]					
	Specimen G91			Specimen G71		
	Calibration Run[a]	Specimen in Place	Elongation	Calibration Run[a]	Specimen in Place	Elongation
0.0	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
0.5	0.0 (0.0)	3.8 (1.5)	3.8 (1.5)	−3.8 (−1.5)	0.0 (0.0)	3.8 (1.5)
1.0	0.0 (0.0)	3.8 (1.5)	3.8 (1.5)	−5 (−2.0)	1.3 (0.5)	6.4 (2.5)
1.5	0.0 (0.0)	3.8 (1.5)	3.8 (1.5)	−6.4 (−2.5)	1.3 (0.5)	7 (3.0)
2.0	0.0 (0.0)	3.8 (1.5)	3.8 (1.5)	−7 (−3.0)	1.3 (0.5)	8.9 (3.5)
2.5	−1.3 (−0.5)	3 (1.0)	3.8 (1.5)	−7 (−3.0)	1.3 (0.5)	8.9 (3.5)
3.0	−1.3 (−0.5)	3 (1.0)	3.8 (1.5)	---	1.3 (0.5)	---
3.5	−1.3 (−0.5)	3 (1.0)	3.8 (1.5)	---	1.3 (0.5)	---
4.0	---	3 (1.0)	---	---	1.3 (0.5)	---
4.5	---	3 (1.0)	---	---	---	---

[a]Calibration runs made with steel blank in place.

installed and allowed to equilibrate. Then the water was added again, the system sealed, and measurements taken. The system is capable of length measurement with an uncertainty of $\pm 1 \times 10^{-5}$ mm ($\pm 0.5 \times 10^{-6}$ in.).

Room temperature was carefully controlled to 20°C ±1°C (68°F ±1°F) and relative humidity was 43% for the swelling measurements. Calibration runs were made to account for the effect of evaporation on the equipment after distilled water was added. Readings

were taken every half hour for the two specimens for several hours. The data are reported in Table IX. After subtracting the calibration curve for each specimen, the maximum amount of swelling is 0.0012%. This is consistent with the data reported for the Oconee-2 flakes. The measurement uncertainty is estimated to be $\pm 1 \times 10^{-5}$ mm ($\pm 0.5 \times 10^{-6}$ in.).

CONCLUSIONS

Based on the data acquired thus far, several conclusions can be made. The magnetite flakes were found to contain particles in the 0.003-mm (10^{-4}-in.) size range. In addition, light microscopy revealed evidence of a possible glassy phase. With one exception, all of the flakes studied were either one- or two-layered as judged by abrupt changes in grain size. The thickness of the flakes studied ranged from 0.0470 to 0.108 mm (0.00185 to 0.00427 in.).

The fracture stress of the flake materials tested was in the range of 20.0 to 113.8 MPa (2.9 to 16.5 ksi). There did not appear to be a substantial change in the range of stresses measured at elevated temperatures. There was no evidence of delamination during bend testing.

The thermal expansion data were similar to those obtained for the Oconee flakes. The mean coefficient of linear thermal expansion was a factor of ~2 larger than that of Fe_3O_4. The maximum amount of swelling measured was 0.0012%, which is consistent with the data on the Oconee-2 flakes.

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