P1-92-AR-P1 Microprobe analyses of Fe oxide detrital grains

PI/Data Contact: Dennis A. Darby (Old Dominion University)

Platform ID: (USCGC Healy, 2002) Instrument: core collected by USGS using Jumbo Piston Core; samples analyzed by ETEC electron microprobe with Krisel automation

Piston Core P1-AR-92-P1 Lat.: 73° 42.40'N Long.: 162° 44.60'W Water Depth: 201n Note: Microprobe data corrected to <100.5% using various assumptions depending on mineral Mineral Numbers: 0=fresh ilmenite, 1=altered ilmenite (>51%TiO2), 2= titanomagnetite, 3=hematite, 4=ilmenohematite, 5=ferroilmenite, 6=magnetite, 7=magnetite +other phases, 8=chromite, and 9=other (silicate with Fe oxide inclusions). See Explanation file for alteration degree, exsolution types, and inclusions. Twinned=1, untwinned=0, rounded=1, subrounded to angular=0. Alteration of 0 = fresh and 4 is highly altered Inclusions = 0 (none), 77= random voids or pits on surface of polished grain

Fe oxide mineral grains were extracted from the 45-63 μ m and 63-250 μ m size fractions using first a strong hand magnet and then a Frantz magnetic separator set at 0.3amp. The magnetic grains from both size fractions were combined and mounted in 1 inch diameter epoxy plugs with dividers so that 4 samples are mounted in each plug. The plugs are ground and polished to expose the grains. Mineral identification was then done with a Nikon reflected light microscope using 100X emersion objective and 10X eyepiece (enlargement = 1000X).

The mineral identification was later confirmed by the microprobe chemistry.

Mineral alteration was noted according to stages where 0=unaltered, to 4=highly altered.

Mineral exsolution if any and type is noted see below for key to various types of exsolution shape.

Inclusions were also noted. See below for key of types noted.

Twinning was also noted where 0=no twinning and 1=twinned.

Rounding was designated as either not well rounded (0) or well rounded (1).

Oxide abundances are in percent.

Fe oxide grains extracted from P1-AR-92-P1were analyzed using an ETEC Autoprobe, an electron probe micro analyzer (EPMA), operating in wavelength dispersive mode (WDS) with an accelerating voltage of 15 kilovolts, a beam current of 230 picoamperes, and a beam diameter of approximately 20 μ m. Peak and background count times were set at 20 seconds or 20,000 counts whichever came first.

All Fe-oxide grains were analyzed by the EPMA for TiO₂, FeO, MnO, MgO, SiO₂, Al₂O₃, Cr₂O₃, V₂O₃, CaO, ZnO, Nb₂O₅, and TaO content (Bence and Albee, 1968). The interference of the Ti K β X-ray emission peak with the V K α peak was corrected by the proportional overlap method developed by Snetsinger and others (1968).

Lithium fluoride (LIF), pentaerythritol (PET), and thallium acid pthalate (TAP) diffracting crystals were used in the ETEC Autoprobe along with argon-filled detectors to collect the characteristic X-rays needed for the elemental analysis. Mineral standards were obtained from the Smithsonian Institution except for the standards used for V, which was precipitated from reagent grade V_2O_5 . Artificial LiO crystals of Nb and Ta was used for these element standards. The chemical compositions of the ilmenite (USNM

96189), anorthite (USNM 137041), chromite (USNM 117075), and gahnite (USNM 145883) standards are published in Jarosewich and others (1980).

Ilmenite was used as a standard for Ti, Fe, and Mn, vanadium pentoxide was used as a standard for V, lithium tantalate was used as a standard for Ta, and Gahnite was used as a standard for Zn. The characteristic emission X-rays for those elements were diffracted to the detector via a lithium fluoride (LIF) crystal. Lithium niobate was used as a standard for Nb, anorthite was used as a standard for Ca, and chromite was used as a standard for Cr. The characteristic emission X-rays for those three elements were diffracted to the detector via a standard for Ca, and chromite was used as a standard for Cr. The characteristic emission X-rays for those three elements were diffracted to the detector via a pentaerythritol (PET) crystal. Anorthite was used as a standard for Si, chromite was used as a standard for Mg, and gahnite was used as a standard for Al. The characteristic emission X-rays for those three elements were diffracted to the detector via the thallium acid pthalate (TAP) crystal.

References:

Jarosewich, E.J., Nelen, J.A., and Norberg, J.A., 1980, Reference samples for electron microprobe analyses. Geostandards Newsletter 4, 257–258.

Bence, A.E., and Albee, A.L., 1968, Empirical correction factors for the electron microanalysis of silicates and oxides: Journal of Geology, v. 76, p. 382-403.

Snetsinger, K.G., Bunch, T.E., and Keil, K., 1968, Electron microprobe analysis of vanadium in the presence of titanium: American Mineralogist, v. 53, p. 1770-1773.

Mineralogy codes

- 0= "Fresh" Ilmenite (Fe⁺²TiO₂): Ilmenite that has not been altered
- 1 = Ilmenite (Fe⁺²TiO₂): includes Ilmenite w/ inclusions but not exsolution features, R % 17-20 (≤ Magnetite), Reflectance/Color in oil = Low/light brown somewhat pinkish-violet, pleochroic in oil= moderate (pinkish brown to dark brown), twins common (10bar11), Strong anisotropism = light greenish gray, brownish gray, (FYI: VHN=659-703; VHN = Vickers hardness number)
- 2= Titanomagnetite: R \leq Ilmenite, slightly anisotropic, easily confused with stage 1 altered Ilmenite
- 3= Hematite (α Fe₂O₃): R%=26-30 (> Ilmenite), Reflectance/Color in oil = Medium/bluish gray, pleochroism in oil = weak, twins very common (10bar11), moderately anisotropic (grayish blue, grayish yellow, greenish gray, light brown tints), red to bright red internal reflections are characteristic (very common as exsolution lenses in Ilmenite or Magnetite), VHN 1038

- 4= Titanohematite: Hematite > Ilmenite
- 5= Ferroilmenite: Ilmenite > Hematite
- 6= Magnetite $(Fe^{+2}Fe_2^{+3}O_4)$: R%=20, Reflectance/Color in oil = Medium/gray, commonly with brownish tint, non-pleochroic in oil, twinning – commonly lamellar (111), isotropic, internal reflections only if Mn-rich, VHN=592
- 7= Magnetite with Ilmenite, Hematite, or Spinel = indicate Mag + Ilm, Mag + Hem or Mag + Spinel
- 8= Chromite (Fe⁺²Cr₂O₄): R%=12.3, Reflectance/Color in oil = Low/dark gray to brownish gray, non-pleochroic in oil, twinning not present, isotropic, internal reflections common = brown to red-brown in Mg-Al rich samples but absent in Fe-rich samples, VHN=1332
- 9= Other mineral, usually a silicate with some Fe oxide inclusion

Mineral Alteration

- 0 = Stage 0, unaltered, highly pleochroic, anisotropic, **R**eflectance = 17 %
- 1 = Stage 1 alteration, weak pleochroism, weak anisotropic, no internal reflection, Reflectance = 18 %
- 2 = Stage 2 alteration, non-pleochroic, no internal reflection, gray efflorescence,

Reflectance = 19 %

3= Stage 3 alteration, non-pleochroic, brown or reddish-brown internal reflections, light gray spots of cryptocrystalline TiO₂ ("cloudy" appearance),

Reflectance = 20 %

4= Stage 4 alteration, yellow or white internal reflections, "cloudy" appearance, Reflectance = 21-22 %

Modified Last: 02/17/2010

Flasers are sigmoidal shaped lenses. Rods are elongated cylinders.

Exsolution style

(Use only on minerals 4, 5 and 7)

- 00 = NONE
- $03 = flasers < 1\mu \& 1-5\mu$
- $05 = \text{lenses}(>5\mu) + \text{flasers}(<2\mu)$
- 06 =lenses (>5 μ) + blebs elongated (<1 μ)
- 07 =lenses ($<5\mu$) + flasers ($<1\mu$)
- 08 = Fracture Pattern
- $10 = straight \ laminae > 5\mu$
- $11 = \text{straight lam } (>5\mu) \text{ in } 2 \text{ directions}$
- $12 = \text{straight lam} (>5\mu) \text{ in 3 directions}$
- $13 = \text{straight lam} (>5\mu) \text{ in } \ge 4 \text{ directions}$
- 14 =lenses in 2 directions
- 20 =thin straight laminae, 1-5 μ
- 21 =thin straight lam (1-5 μ) in 2 directions
- 22 =thin straight lam (1-5 μ) in 3 directions
- 23 = thin straight lam $(1-5\mu)$ in ≥ 4 directions
- 24 = very thin straight laminae, $<1\mu$
- 25 = very thin straight lam in ≥ 2 directions
- $26 = \text{combo of straight laminae}, <1 \& 1-5\mu$
- $27 = \text{combo of straight laminae}, <1 \& >5\mu$
- $28 = \text{combo of straight laminae}, 1-5 \& >5\mu$
- $29 = straight \ laminae \ \& \ flasers, < 2\mu$
- 30 =lenses $> 5\mu$ wide
- 31 =lenses $1-5\mu$ wide
- 32 =lenses $< 1\mu$ wide
- $33 = \text{combo of} < 1 \& 1-5\mu \text{ wide lenses}$

- $34 = \text{combo of } <1 \& >5\mu \text{ wide lenses}$
- $35 = \text{combo of } 1-5 \& >5\mu \text{ wide lenses}$
- 36 = combo straight lam & lenses, any thickness
- 37 =lenses (<1 μ) + rods (<1 μ)
- $38 = \text{lenses} (\geq 1\mu) + \text{rods} (\geq 1\mu)$
- 39 = wedge only
- 40 = wedges $> 10\mu$ at widest point
- 41 = wedges & straight laminae $<1\mu$ within
- 42 = wedges & straight laminae 1-5 μ within
- 43 = wedges & straight laminae > 5 μ within
- 44 = wedges & lenses $<1\mu$ within
- 45 = wedges & lenses 1-5 μ within
- 46 = wedges & lenses > 5 μ within
- 47 = wedges & flasers $<1\mu$ within
- 48 = wedges & flasers 1-2 μ within
- 49 = wedges & flasers >2 μ within
- 50 = flasers (sigmoidal lenses) $< 1\mu$
- 51 =flasers $1-2\mu$
- $52 = \text{flasers} > 2\mu$
- 53 = flasers & straight laminae $<1\mu$ within
- 54 = flasers & straight laminae 1-5 μ within
- 55 =flasers $<1\mu$ in two directions
- 56 = flasers 1-2 μ in two directions
- 57 =flasers $> 2\mu$ in two directions
- $58 = rods (< 10\mu)$ width
- $59 = rods (>10\mu)$ width
- $60 = \text{irregular blebs (elongated)} > 1 \text{ or } 2\mu$

- 61 = irregular blebs (elongated) ≤ 1 or 2μ
- 62 = irregular blebs (random orientation & shape) >1-2 μ
- 63 = irregular blebs (ran orientation & shape) $\leq 1-2\mu$
- 64 = irregular blebs (elongated) in two direct
- 65 = irregular blebs (elongated) in ≥ 3 directions
- $66 = \text{straight lam} (>5\mu) \text{ w/ irregular blebs within}$
- $67 = \text{straight lam } (1-5\mu) \text{ w/ irregular blebs within}$
- 68 = wedges with irregular blebs within
- 69 = flasers ($\geq 2\mu$) & irregular blebs

Inclusion Type & style

00 = none

- $70 = straight lamellae voids < 1\mu$
- $71 = \text{straight lamellae voids } 1-5\mu$
- $72 = \text{straight lamellae voids} > 5\mu$
- 73 = flaser voids any size
- $74 = lensoid voids < 5\mu$
- 75 = lensoid voids $\ge 5\mu$
- 76 =rod-shaped voids any size
- 77 = irregular voids any size
- 80 = any silicate
- 90 = apatite
- 91 = spinel (isotropic)
- 92 = ulvospinel
- 93 = ulvospinel and straight laminae
- 94 = ulvospinel and lenses
- 95 = ulvospinel and flasers
- 96 = ulvospinel and wedges
- 97 = ulvospinel and voids

98 = other inclusion and exsolution

99 = unknown inclusion

Ulvospinel = (ulvite): color browner than Magnetite. Key to ID is that texture is extremely fine and occurs as exsolutions in Magnetite.