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Microbeam Analysis of Plasma Effects in Synthetic Mica-Like Compound

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The effect of argon plasma exposure in a synthetic mica-like compound was studied by SEM, TEM and EMPA. The material studied was a laminate mica paper from McMaster-Carr, part number 8779K11. The energy of the electrons in the plasma was 40-80eV with a current density greater than 1A/cm^2 for 4-6 hours. The original compound was transparent silver. The plasma exposed mica forms a concentrated dark line in the focused beam region and slight darkening of the overall sample as shown in Fig. 1.

The mica-like samples were examined in polished sections by optical and scanning electron microscopy (SEM, Hitachi S3200N equipped with energy dispersive X-ray spectrometry, EDS).The chemical composition of the compound was quantitatively determined by an electron microprobe analyzer (EMPA, Cameca SX100). The accelerating voltage and beam current were 15 kV and 10 nA, respectively with 1 μm beam diameter. The counting times on the peak were 30 seconds with half of that time on both sides of the peak. The PAP correction procedure was used for the analyses.

The back-scattered electron (BSE) images of the compound treated by plasma in a section perpendicular to the perfect cleavage planes (001) show that it contains two main phases: (i) The "brighter"-phase in BSE contrast, which reflects higher average atomic weight; and (ii) The "darker"-phase in BSE, which occurs between the crystals of phase-(i) (Fig. 2a). Phase-(ii) occurs near the plasma treated surface. Phase-(i) contains Fe-oxide impurities <0.5 μm in size. The semi-quantitative EDS analyses reveal that phase-(ii) is significantly depleted with Al, K and Fe compared to phase-(i) (Fig. 2b,c). EDS in both the SEM and TEM revealed the same qualitative reduction in Al, K and Fe after plasma exposure. The chemical compositions of the untreated and plasma-treated compounds are given in Table 1. The EMPA elemental mapping revealed inhomogeneous distribution of Fe and Al, as well as the occurrence of Ca-S rich impurities (Fig. 2a). Phase-(ii), which was found in the interstitials of phase-(i) is enriched with SiO_2 and depleted with Al_2O_3, K_2O and FeO compared to phase-(i) and the untreated "mica." The analytical totals of phase-(ii) are lower by ~10-20wt% of oxides than the other phases (Table 1).

The EMPA analyses revealed that the chemical composition of the untreated sample and phase-(i) is similar (Table 1), which confirms the thermal stability of the studied compound. However, plasma exposure leads to separation of phase-(ii). There is a relative increase in the concentrations of SiO_2, 18-29%; and loss of Al_2O_3 and K_2O varying in range from 57-83%, 43-81%, respectively, between the untreated compound and separated phase-(ii). The compositional changes caused by the plasma exposure are an interesting effect but do not explain the observed color change. The color change is due to a change in oxidation state on the iron [1]. Mossbauer spectroscopy will be performed to quantify the oxidation state change.

References

[1] Kurt Nassau, *The Physics and Chemistry of Color – The Fifteen Causes of Color*, John Wiley & Sons, Inc., New York, 1983.

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Figure 1. Mica-like compound showing dark lines caused by plasma exposure.

Table 1. Chemical composition of mica-like compound determined by EMPA.

	Not treated	Treated phase-(i)	Treated phase-(ii)
Na_2O	0.21-0.80	0.28-0.87	0.04-0.30
MgO	0.17-0.63	0.18-0.93	0.02-0.20
Al_2O_3	33.12-37.46	29.38-35.94	4.17-16.07
SiO_2	44.52-51.73	46.75-52.98	54.29-74.40
K_2O	6.08-10.00	6.40-9.63	1.15-4.33
FeO	1.27-3.84	1.47-3.18	0.33-1.09
Total	93.22-98.87	90.90-97.32	76.31-80.12

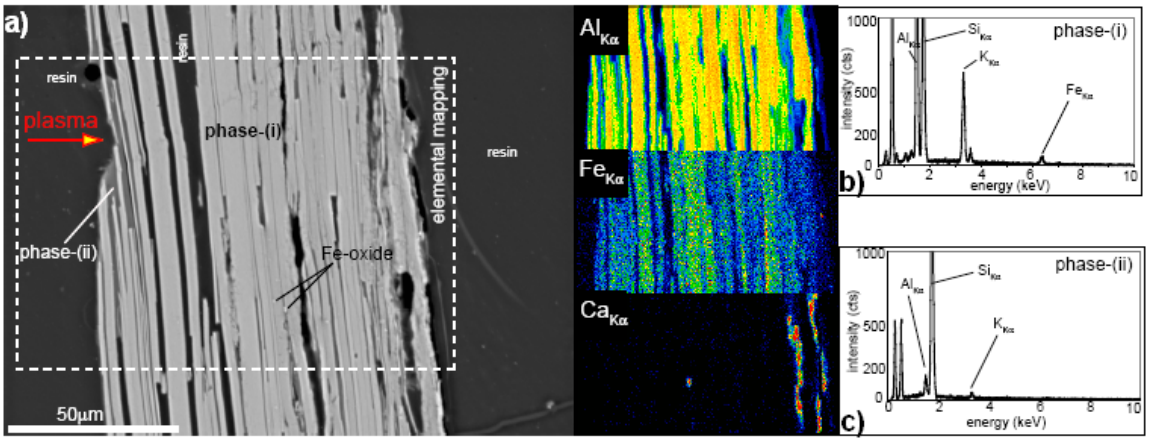

Figure 2. a) BSE image and EMPA elemental maps of cross-sectioned mica-like compound after plasma treatment. Note two phases (i) and (ii), which differ in BSE contrast. b-c) EDS spectra of phases (i) and (ii), respectively; note relative decrease in Al, K and Fe concentration in phase (ii).

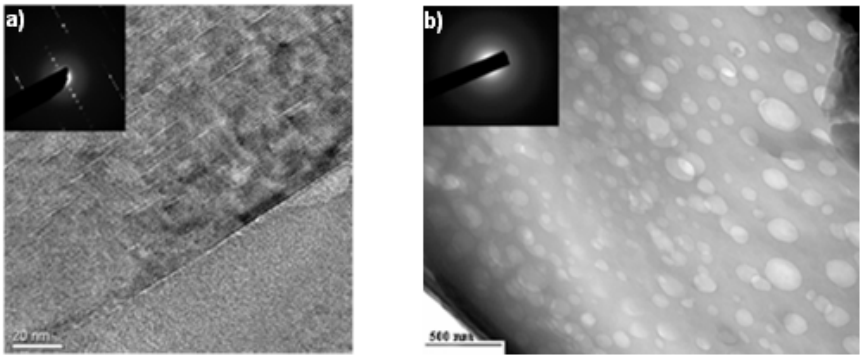

Figure 3. Cross-sectional TEM micrograph showing both crystalline (a) and amorphous (b) regions of sample. Bubbles shown in b) result from electron beam irradiation.