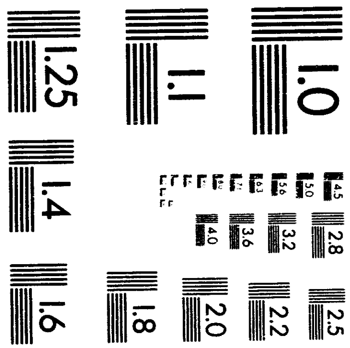

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CONTINUUM FLUORESCENCE OF Cu IN NbTi/Cu COMPOSITE WIRES

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Continuum fluorescence of characteristic x rays, at an intensity sufficient to produce serious errors in quantitative x-ray microanalysis, is rarely encountered in the electron probe microanalyzer (EPMA). The physics of fluorescence produced by characteristic and continuum x-rays are identical except for the source of the fluorescing radiation. Continuum fluorescence has been included in some correction programs, but these only apply to fluorescence that occurs in a homogeneous material.[1] Continuum fluorescence across interfaces separating regions of differing composition is difficult to calculate rigorously. This paper illustrates a case of continuum fluorescence in the analysis of superconducting NbTi/Cu composite wire which could lead to erroneous compositional information due to the fluorescence of Cu by continuum x rays generated in an NbTi alloy. An approximate treatment of the continuum fluorescence will be presented.

Wires for superconducting magnets are produced by alternately drawing and annealing of billets assembled from NbTi alloy rods sheathed in Cu. The drawing and annealing operation is continued until the composite wire reaches the desired size. The NbTi alloy is surrounded by sheaths of Nb as a diffusion barrier to prevent the diffusion of Cu into the superconducting NbTi alloy.[2] The Nb diffusion barrier is necessary to prevent Cu intermetallic compounds from forming at the Cu/NbTi interface, which can cause wire failure during drawing.[3] During an EPMA study of the effectiveness of the Nb diffusion barriers, it was noted that all of the wires showed unexpected high levels of Cu in the NbTi filaments. Examination of samples from different stages in the processing, including drawn but not heat-treated wire, showed essentially identical apparent Cu penetrations into the NbTi filaments to a depth of approximately 50 μm. After checking for a variety of possible artifacts, including smearing during metallographic preparation, beam flare, etc., it was concluded that fluorescence of the Cu Kα x ray by the continuum generated in the NbTi filament was the most probable cause of the false Cu signal.

EMPA wavelength dispersive spectrometry (WDS) was carried out using an accelerating voltage of 15 kV. Compositional profiles were measured across the Cu/NbTi composite specimens using first the Cu Kα line and then the Cu Lα line. Figure 1 shows the profile obtained when the Cu Kα line was used for quantification. The elevated Cu signal is apparent in the NbTi alloy. Figure 2 shows a similar profile obtained using the Cu Lα line. Comparison of Figures 1 and 2 clearly shows that the elevated apparent Cu level found when the Cu Kα line was used is not present when Cu Lα radiation was used.

The intensity of continuum fluorescence generated in the specimen is evaluated in a similar manner to the corrections for characteristic fluorescence, with the exception that the fluorescing radiation is polychromatic for continuum fluorescence. An integration was performed from the critical excitation voltage of the Cu Kα or the Cu Lα lines to the beam voltage. This integration included terms to describe the intensity of the continuum generated in the NbTi alloy, the ionization cross section for the Cu line of interest as a function of x-ray energy, and a term to account for the absorption of the continuum x rays as a function of x-ray energy and distance into the NbTi. Figure 3 shows the results of these calculations. The data were normalized so that the relative differences in the apparent depth of Cu penetration could be observed when different Cu signals were utilized. It is apparent that the continuum fluorescence effect is large when the Cu Kα is used. This effect is a result of the minimal amount of absorption of the fluorescing radiation with an energy greater than the critical excitation energy of Cu Kα (8.98 keV). The

Cu Lα x rays are not as highly fluoresced because the critical excitation energy for Cu Lα is about 0.93 keV, and x rays that are efficient at fluorescing Cu Lα x rays are heavily absorbed within the NbTi and the intensity that propagates to the Cu is quite low.

These experimental results, combined with the calculations, reinforce the need for careful selection of x-ray lines for quantitative analysis when compositional information is required from a sample with a heterogeneous microstructure. The quantitative microanalysis of the superconducting NbTi filaments demonstrated that the continuum generated in the NbTi had sufficient intensity over the distance to the Cu sheath to fluoresce the Cu Kα line, resulting in an apparent Cu concentration in the NbTi. The continuum generated in the NbTi did not have sufficient intensity to significantly fluoresce Cu Lα x rays in the Cu sheath resulting in an accurate determination for the composition of the NbTi filaments.[4]

References
1. K. F. J Heinrich, *Electron Beam X-ray Microanalysis*, New York:Von Nostrand Reinhold,(1981)328.
2. M. T. Taylor, et al., *Cryogenics*, June 1971.
3. J. M. Seuntjens, private communication, May, 1993.
4. This work was supported by the U. S. Department of Energy under contracts # DE-AC04-94AL85000 and DE-AC02-89ER40486.

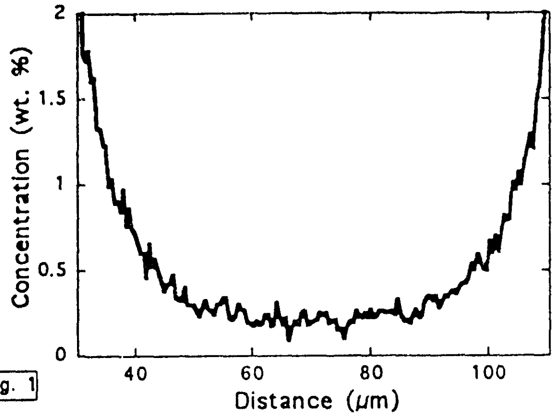

Fig. 1

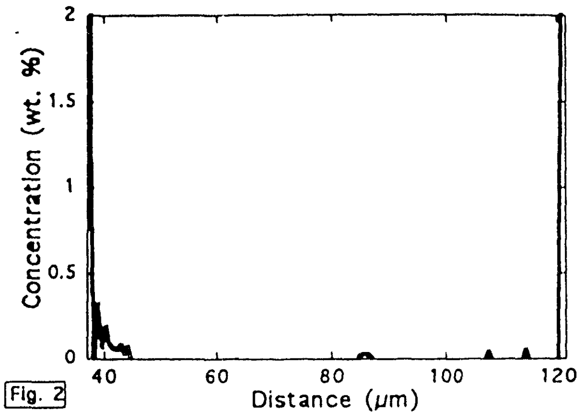

Fig. 2

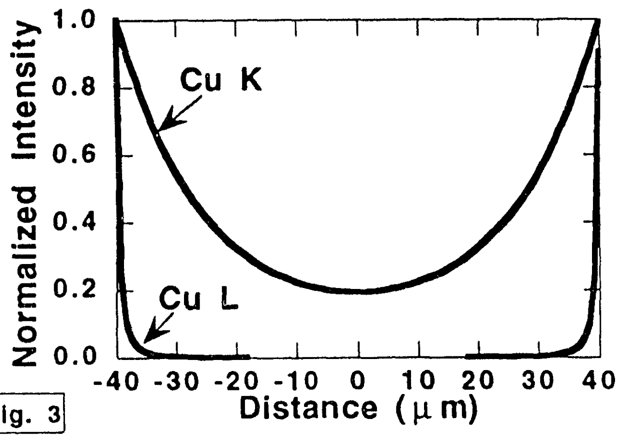

Fig. 3

Fig. 1. Cu EPMA composition profile across a NbTi filament from the Cu Kα x ray line.
Fig. 2. Cu EPMA composition profile across a NbTi filament from the Cu Lα x ray line.
Fig. 3. Calculated normalized profiles of continuum fluoresced Cu showing the effect on the Cu Kα line and the Cu Lα line.

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