Electron Microscopy and EDS of Nanoparticles Generated During Friction Stir Welding

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In this paper we present a brief summary of the characterization of friction stir welding (FSW) [1] aerosols by electron microscopy (EM) and energy dispersive spectroscopy (EDS).The motivation for this work was to characterize possible exposures of students and researchers to engineered nanoparticles in a University laboratory setting. FSW, an active process in the lab, was a good case to study because little is known of emissions from FSW and preliminary data indicate fine aerosol emission. This paper is one of a series [2,3] from a larger comprehensive aerosol study that used a suite of instruments to adequately characterize size distribution, morphology, and chemical composition of fine and ultrafine aerosols. Significant source emissions (10^5 to 10^7 pts/cm^3) of nanometer and submicrometer particles were observed, most of which consisted of Al, Mg, Zn, and Fe. EM is an indispensable tool in the characterization of aerosols, especially morphology, agglomeration, surface area, and elemental composition, all of these being important parameters of particle toxicity. In addition the EM data can be directly compared with and validated against other independent measurements of fine aerosol properties.

Airborne particles were collected directly onto 3 mm Cu grids with 10 nm SiO support films using a point-to-plane corona discharge Electrostatic Precipitator (ESP) placed 20cm from the weld (source) and a Thermophoretic Precipitator (TP) located in the operator's breathing zone (BZ, 92 cm from weld). Instruments were placed side-by-side with a larger suite of real time aerosol monitoring instruments. The grids were mounted in a special holder for examination in the LEO 1530 FESEM at the UW, and into a standard STEM holder for work in the Hitachi S-5500 SEM/STEM at RJ Lee Group. EDS spectra were collected for 300 seconds at 20 kV with a 10 mm^2 silicon drift detector (SDD) at a 35 degree takeoff angle.

Secondary electron (SE) images of representative fields from the source and BZ were taken at a magnification of 4000X (Fig. 1). The particles range in size from 10 nm to greater than 2 μm in diameter. A series of images were taken at 4,000X and analyzed with IMAGEJ [4] to determine the particle size distribution. The distribution peaks in the 60 to 85 nm range with a long tail out to several microns (Fig. 1) and compares favorably with the real time size distribution. A variety of chemistries were found by EDS, but both large and small particles were found that contained Mg and Al, the major constituents of the 6061-T6 Al alloy work piece (Fig. 2). These observations agree well with the size-selective chemical analysis using inductively coupled plasma mass spectrometry. The particle morphology suggests the agglomeration of smaller particles that appear to have melted or bonded together to form larger particles, while larger particles have a chip like morphology (Fig. 2).

The results of the study show that FSW produces significant emissions of nanometer and micrometer size particles from the work piece at a level several orders of magnitude above the laboratory background level. The EM analysis provides invaluable evidence about the morphology of particles and agglomeration dynamics and should be a requirement for studies of nanoparticles emissions.

This comparative work suggests that good quality stand-alone EM work can provide reliable information on particle size distribution and chemistry of aerosols.

References:

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[2] F. E. Pfefferkorn et al, *submitted to Journal of Aerosol Science and Technology*, (2009)
[3] J. J. McCarthy, *submitted to MAS Topical Conference on Microanalysis of Particles*, (2009)
[4] M.D. Abramoff, P.J. Magelhaes, and S.J. Ram, *Biophotonics International.* 11 (2004) 36

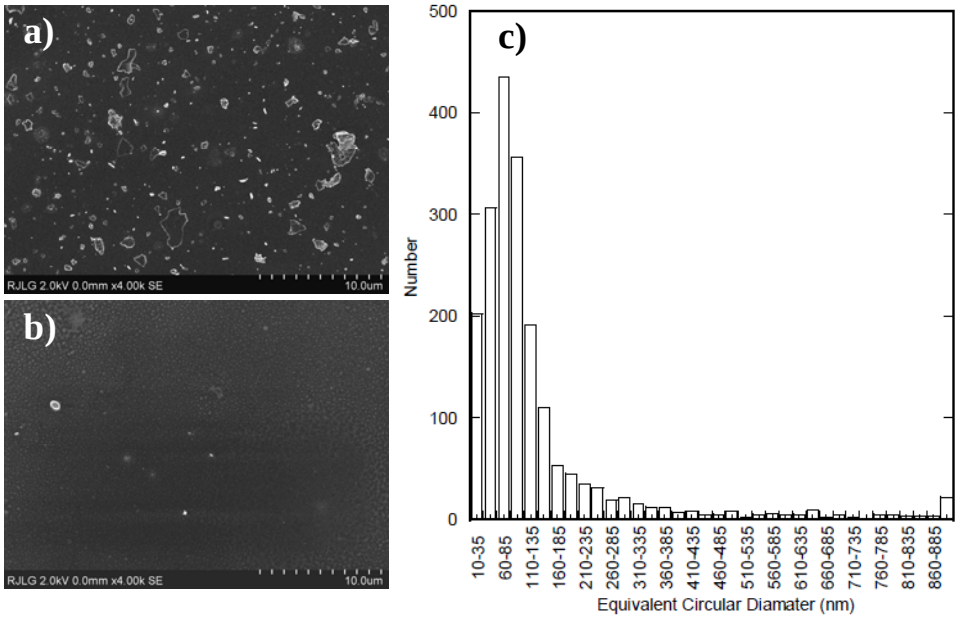

Fig. 1: Secondary electron images of representative fields collected a) with ESP at the source and b) with TP at the breathing zone and c) a particle size distribution for the ESP.

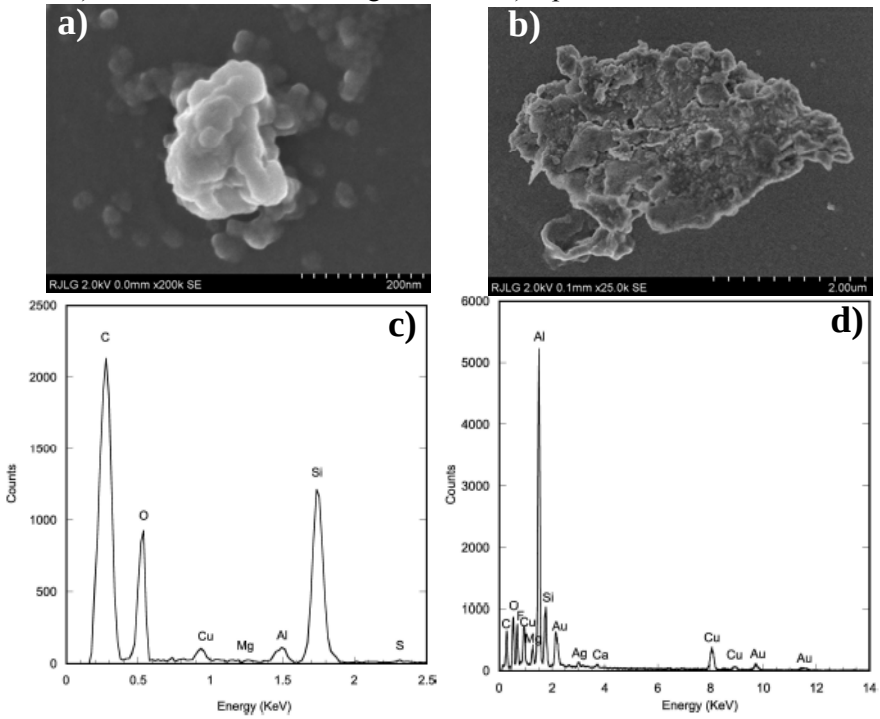

Fig 2: Secondary electron images of a) a 150 nm agglomerated particle and b) a larger particle collected at the source with c) and d) corresponding EDS spectra showing Mg and Al.