Evaluating Expanded Polymers Using a Variable Pressure Large-Chamber Scanning Electron Microscope

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The Y-12 National Security Complex in Oak Ridge, TN operates a large-chamber scanning electron microscope (LC-SEM) that has the largest chamber in the world at eight cubic meters. This microscope provides high-resolution images at magnifications from 10x to 200,000x, while using a mobile column that moves around the sample on a four-axis positioning system. In addition to the most extensive array of analytical equipment of any SEM in the world, the LC-SEM at Y-12 is equipped with a variable pressure (VP) mode that allows the evaluation of non-conductive samples without the need of sputter-coatings.

Using the VP mode of the LC-SEM has enabled the evaluation of bonding properties of expanded polymer beads. It has been well known in the expanded polystyrene (EPS) and related industries that the properties of EPS resulting from the dual expansion and molding process can vary greatly according to the process conditions and starting bead. However, it has been difficult to relate the processing of the beads and bonding to the final mechanical properties of the part. These mechanical properties are important in products such as bicycle helmets, concrete wall supports, and lost-foam casting molds.

This effort is an attempt to relate the raw bead properties and the molding processing characteristics to the mechanical consequences of the resulting part. We believe that the pre expanded bead has a critical impact in the macro-mechanical properties of the resulting molded part. For example, the smoother surface of the raw bead allows for better contact with the neighboring beads upon expansion, creating a stronger bonding capable of withstanding greater mechanical stresses.

The VP mode has been studied to determine favorable operating conditions to allow optimal image quality. High voltage, current, working distance, and chamber pressure were variables considered when creating an image from the four-quadrant backscatter electron (BSE) detector. The same quadrants were used to create each image, though brightness and contrast adjustments were necessary. Fourier transform infrared (FT-IR) in the LC-SEM was used to evaluate the chemical properties.

In addition to chamber pressures, the paths to achieve these pressures were evaluated. Some expanded polymers do not maintain their structural integrity as well as others under vacuum conditions. Thus, the chamber pressure was not only evaluated on its effect of image quality, but also how the pressure directly visualized the structural and mechanical properties of the polymer. In order to lessen the stresses imposed on the expanded structures, higher vacuum pressures were attempted straight from atmospheric pressures, thus bypassing higher vacuums. Chamber pressures ranging from 1e-2 mbar and higher were evaluated.

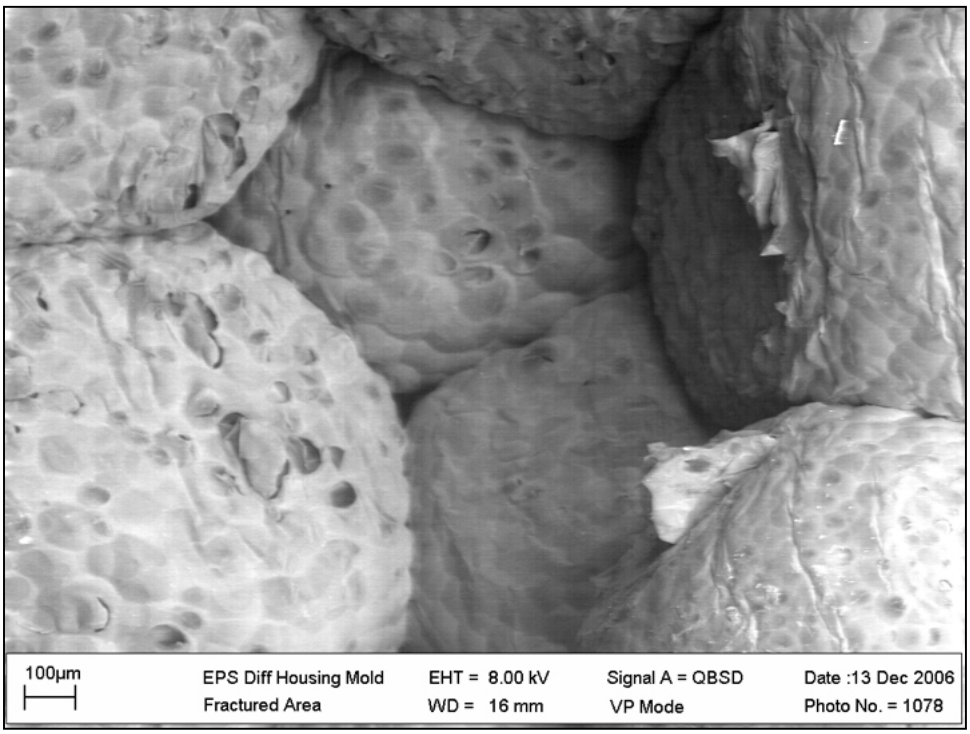

Figure 1: SEM image (200x) of fractured EPS showing separation of the still intact beads.

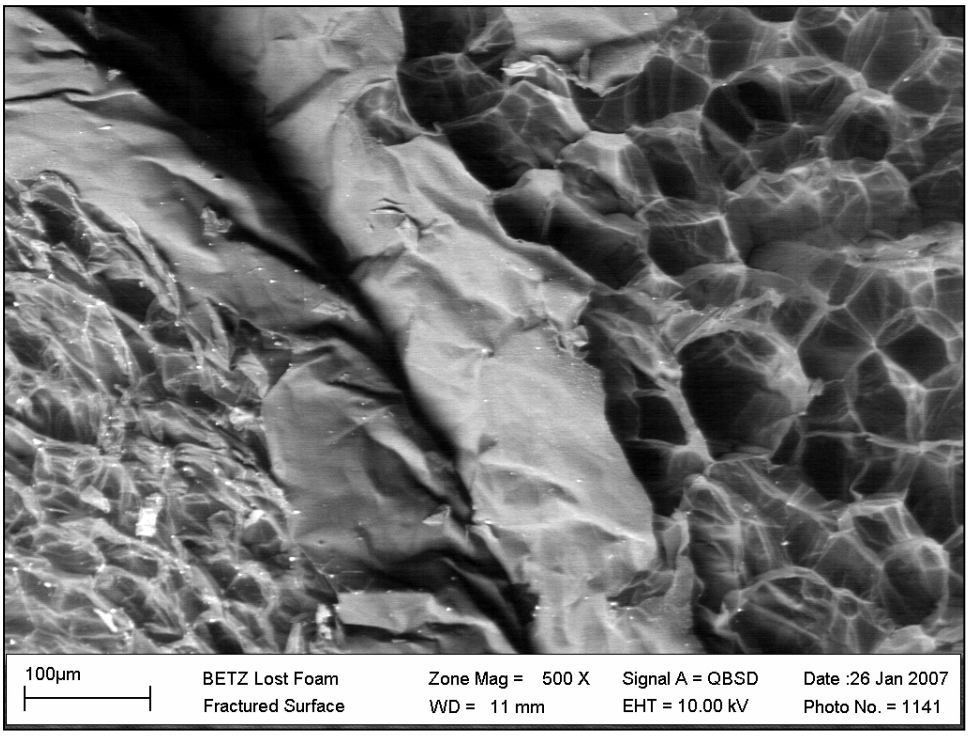

Figure 2: SEM image (500x) of fractured EPS showing fracture within the bead while maintaining the bead joint.