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A particulate-sample mounting technique for the laser Raman microprobe

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The laser Raman microprobe utilizes the power of conventional Raman spectroscopy for fingerprint phase identification and the magnification capabilities of the optical microscope. The micro–Raman spectroscopic method allows the examination of samples of the dimension of the diffraction limit of a focused laser beam, approximately 1 μm. Sample preparation is generally very simple; a mineral sample in thin section or thick section can be viewed in either reflected or transmitted light, or powders can be dispersed onto a conventional microscope slide. With this approach, compositions of fluid inclusions can be measured (Pasteris et al., 1985; Wopenka and Pasteris, 1986) as well as accessory mineral phases in thin section or aerosol particles. Scheetz et al. (1985) and Scheetz and White (1985) applied this apparatus to the characterization of both crystalline and glassy components of fly ash from coal-powered electrical-generation facilities.

Because the laser beam is directed through the optical pathways of a light microscope, the laser light is subject to all of the instrumental constraints of resolution that are imposed upon a conventional microscope. A consideration that significantly impacts the intensity of the Raman signal is light scattering from small particles with an air interface. The use of a fluid-immersion lens system to minimize this type of signal loss was described by Andersen and Muggli (1981). Traditional oil-immersion lenses and oils are not satisfactory for Raman application because of the fluorescence that is generated by the intense laser radiation interacting with the organic immersion oils.

A survey of mounting media that would retain dispersed fine particulate materials for examination with the laser Raman microprobe and would not contribute detrimental fluorescence yielded several suitable candidates. These included collodion, as suggested by Andersen and Muggli (1981); cellulose acetate; cyanoacrylate ester (super-glue); Duco cement, and 3M Photo-Mount. In general, any polymeric system composed of saturated aliphatic hydrocarbons will meet the criterion of nonfluorescence.

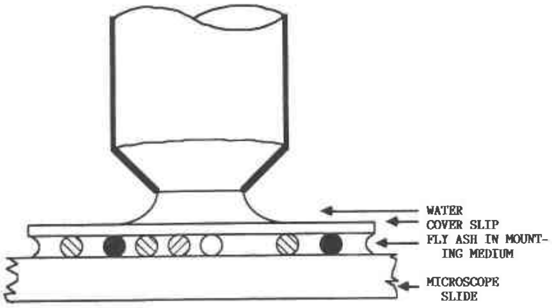

Fig. 1. Schematic drawing of the particulate-mounting method.

A mounting medium is introduced that will be applicable to those investigators who require a high water content (~85%) as the immersion medium for micro–Raman observation of their samples. This particular medium is an extensively cross-linked acrylanide polymer that offers the added advantage of the ability of the investigator to control the hardness of the set polymer ranging from a rubbery to a brittle form. The polymer chemistry is similar to that used for modern extended-wear soft contact lenses, and the specific formulation recommended here is also used extensively to prepare gels for electrophoresis. The polymer is a two-part system in which *N,N'*-methylenediacrylamide (BIS) is used to cross-link acrylamide in the presence of tetramethylethylenediamine (TEMED) and ammonium persulfate (AP). The latter two compounds are used as catalyst and initiator, respectively. Rates of reaction are controlled by varying the concentration of TEMED and AP used, and the viscoelastic stiffness of the resulting gel is controlled over a wide range by varying the proportion of BIS to acrylamide. Table 1 details four different mixing proportions that can be used to tailor the cross-linked polymer from a rubbery to stiff consistency.

The sample preparation that was followed in this approach was identical to a grain mount where the polymer was allowed to cure (Fig. 1). A drawback of this polymer

Table 1. Mix proportions for acrylamide mounting medium

	Rubbery formulation	⟷		Stiff formulation
Constituents	1	2	3	4
Acrylamide (3.0*M*)	47.8	47.6	47.4	47
N,N'-methylenediacrylamide (0.2*M*)	0.2	0.4	0.6	1
Tetramethylethylenediamine (100 μL/mL)	1	1	1	1
$(NH_4)_2S_2O_8$ (50 mg/mL)	1	1	1	1

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system is a loss of water that occurs within 48–72 h, even from beneath the glass cover slip. The use of the harder formulation from Table 1 may result in a slight extension of that time. Gel time may be controlled to allow for longer working times by reducing the concentration of initiator (ammonium persulfate).

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