Cryostat for low temperature x-irradiation and electron paramagnetic resonance

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Abstract. A cryostat for x-irradiation of solid samples at liquid air temperature is described. The design of the sample mount in the cryostat enables quick transfer of the sample after x-irradiation into EPR cavity to facilitate EPR study without warming the sample.

Keywords. X-irradiation; electron paramagnetic resonance; cryostat.

1. Introduction

Several paramagnetic defect centres produced in crystals by x-irradiation are not stable at room temperature. It therefore becomes necessary to irradiate the crystals at low temperature and study the EPR without allowing the sample to warm up. The best way to do this would be to irradiate the sample in situ, with the sample already placed in the EPR cavity and held at low temperature. This requires that one should either have a portable x-ray machine which can be brought to the EPR spectrometer or be able to take the cavity system with its low temperature dewar assembly to the x-ray machine. This is not always possible and convenient. The next alternative is to irradiate the sample in a separate cryostat and then quickly transfer the sample to EPR cavity (Poole 1967; Alger 1968). In general both the methods involve irradiation through two to three layers of quartz/glass envelopes of the dewar and/or sample tube with resulting attenuation of x-rays reaching the sample. Also there is a possibility of defect centres produced in the sample tubes/mounts and/or walls of insert dewar which may interfere in the EPR study. In the latter method there is an additional danger of warming up the sample as well as frosting unless extreme precautions are taken. In this paper, we describe a cryostat which enables irradiation at liquid nitrogen/air temperature and a quick transfer of the sample to the EPR cavity for measurements. This is particularly suitable for use with Varian E-257 variable temperature accessory.

2. Description of the cryostat

The cryostat used for the low temperature x-irradiation is the standard cold finger type with a modified sample mount to enable quick transfer of the sample to the EPR
cavity. Figure 1 shows the details of the construction. The sample C is held in position in a groove on the cold finger copper block (B) by means of a spring loaded mild steel plate (D) screwed on the copper block. The tail section (G) of the vacuum jacket is attached by a conventional O-ring seal and is provided with a window F made from a polythene film for x-irradiation. A slot in the mild steel plate allows x-rays to fall on the crystal.