Table 3.4.1.2. *Single-crystal mounting – adhesives*

Adhesive	Temperature range (K)	Comments
Durofix, Duco cement *etc.* (celluloid composition dissolved in organic solvent)	93 to 373	* Dries rapidly
Shellac dissolved in alcohol	< 423	* Correct amount of solvent is critical
Fish glue (*e.g.* Seccotine)	< 423	* Unsuitable for humid atmospheres
Dental cement	93 to 573	Adheres well to glass or asbestos, but not metals
Epoxy resin (epichlorohydrin, *e.g.* Araldite)	93 to 373	* Permanent fixing, fast (minutes) and slow (hours) available. 'Uncured' adhesive, *i.e.* minus hardener, useful for cryogenic mounting
Vacuum grease (*e.g.* Apiezon)	< 473	Can protect crystal from moisture
Silicone high-vacuum grease	< 373	Can protect crystal from moisture
Vaseline		Low temperatures down to liquid helium
Canada balsam	< 333	† Dilute with xylene.
Mixture of wax and resin, ~1:1	93 to 303	†
Aluminium	< 873	Large crystals set in molten metal, irradiate only protruding part of crystal
Aluminium cement	< 1973	Irradiate only protruding part of crystal

* These glues tend to pull in setting and may require adjustment during the drying process. † Useful adhesives if the crystal requires grinding to shape after fixing.

orientation of the fibre (and crystal) on the goniometer head may also need careful alignment.

Many proprietary adhesives can be used (see Table 3.4.1.2), but it should be remembered that adhesives such as epoxy resins are often permanent, and attempts to dismount specimens lead to crystal damage. Some adhesives contain organic solvents that may react with the sample, and others may be X-ray sensitive and deteriorate with exposure. In low-temperature work, some adhesives shrink or become brittle. Ideally, the adhesive should have the same thermal characteristics as the crystal and its mount. An account of how strong stresses on adhesives, typically used to mount single crystals, induced by low and high temperatures is given by Argoud & Muller (1989*a*). The stresses appear to cause anisotropic modifications to secondary extinction, leading to discrepancies in the intensities of symmetry-related reflections. Beeswax and paraffin wax were found to be free from such stresses. Crystals that are sensitive to air can be mounted inside capillary tubes or other containers, as listed in Table 3.4.1.1. A useful summary of the methods available has been provided by Rao (1989). All adhesives and containers will give diffraction patterns, typically comprising diffuse bands, that contribute to the general background, and that may change with ageing. Minimal amounts of adhesive and thin-walled capillaries should be used. If the background diffraction is critical, it is highly recommended that diffraction patterns of the container and/or adhesive are recorded separately as controls.

The morphology of a given crystal will normally dictate the way that it is mounted, particularly for data-collection purposes. Thus, prismatic crystals and needle-shaped crystals are usually mounted with the longest dimension parallel to the fibre, in order to minimize systematic errors due to absorption. Jeffery (1971) and Wood, Tode & Welberry (1985) have described devices for shaping crystals into spheres and cylinders, respectively. A solvent lathe whereby a string moistened with solvent is used to shape the crystal is described by Stout & Jensen (1968).

3.4.1.3.2. *Non-ambient conditions*

As in the case of polycrystalline samples, a number of devices have been described to study single crystals at elevated pressures and at a range of temperatures. The mounting of the sample is very dependent on the device and radiation used. In recent years, the field of high-pressure crystallography has expanded significantly, and several sample holders based on the diamond-anvil cell have been reported for pressures up to 10 GPa. Recent papers include those by Alkire, Larson, Vergamini, Schirber & Morosin (1985) for neutron diffraction, and Malinowski (1987) and Leszczynski, Podlasin & Suski (1993) for X-ray diffraction.