A high accuracy, high resolution, remote control uniaxi, stress apparatus*

M. Gorman, J. Doehler, and S. A. Solin

The Department of Physics and The James Franck Institute, The University of Chicago, Chicago, Illinois 60637 (Received 1 July 1974; and in final form, 5 August 1974)

A uniaxial stress apparatus designed for low-temperature light-scattering experiments is described. It is more compact and potentially more accurate than previous designs. The apparatus can be used to produce a compressive uniaxial force of up to 8.92×10^8 dynes with an estimated accuracy of 1.5% and can operate in a temperature range of 1.2 to 300 K. The design does not restrict this apparatus to light-scattering measurements and it could be easily modified for application to tensile uniaxial stress. This apparatus has been used to observe the splitting of the valley-orbit Raman line in Ge(As) as a function of uniaxial stress along the [110] axis and to successfully apply the highest reported uniaxial stress along the [110] axis in germanium, 23 kilobar.

The application of uniaxial stress to crystals has proved to be an important experimental technique in the study of the properties of solids.¹⁻⁴ A variety of designs of systems capable of applying a compressive uniaxial stress has appeared in the literature.⁵⁻⁹ For instance, Anastassakis⁹ has recently described an apparatus which can apply both tensile and compressive uniaxial stress with the same sample housing. The apparatus to be described below is a modification of a stress cell originally designed by Fritzsche⁵ for piezoresistance measurements and later modified by Pollak and Cardona⁶ for light-scattering measurements.

The stress cell described in this paper differs significantly from the previously mentioned designs. The force is applied by pressurizing a space between concentric bellows and is measured by a force transducer, a commercially available device which measures force directly. This stress cell has a number of advantages. It is a high accuracy, high resolution, remote control device which is more compact and has less friction than is present in other designs. The stress can be applied quickly and smoothly, can be read immediately, and can be reset easily, all without disturbing the optical alignment of the cell. The apparatus is not limited to optical measurements at low temperatures but can be used for any measurement requiring compressive uniaxial stress. The design can also be easily modified to apply tensile uniaxial stress.

A drawing of the stress apparatus is shown in Fig. 1. An isometric view of the sample housing is shown in Fig. 2. The sample (A) is epoxied into brass cups (B, C) which fit closely into holes in both the piston (D) and the cylinder (E). The piston (D) and the cylinder (E) are machined from the same piece of hardenable stainless steel and therefore have the same coefficient of thermal expansion. To reduce friction, piston (D) is machined so that it touches the cylinder (E) at only three points. After being machined, the piston (D) and cylinder (E) are lapped together for a smooth fit. Holes (not shown) are drilled in the cylinder to allow air that might be trapped between the points of contact with the piston to escape. Otherwise, the trapped air would freeze at liquid helium temperatures and introduce significant additional friction between the piston (D) and the cylinder (E). Two slots (F) through which the sample can be irradiated are machined into the cylinder. A carbon resistance thermometer (not shown) is epoxied to the cylinder (E) near the sample.

A pullrod (G) through which the force is applied fits through a hole in the cap (H) and screws into the junction insert (I) which sits in a slot in the piston (D). A crossbar (J), also made of hardenable stainless steel, slides through holes in the cylinder (E) and the junction insert (I) and is held in place by a set screw (K) thereby securing the pullrod (G) to the cylinder (E). The cap (H) is attached to the piston by four #2 socket head screws (N). The cap also screws into the pushtube (L) which supports the entire sample housing. Holes (M), which serve as feedthroughs for electrical connections to various points in the sample housing, are drilled in the pushtube (L) near the cap (H).

The sample size is dictated by the experiment, transmission Raman scattering, by the collection optics, and by the maximum desired stress, 20 kilobar. In the backscattering Raman configuration, the laser beam is focused to a line on the sample by a cylindrical lens. However, in transmission Raman scattering, the beam is focused to a point thereby producing a filament in the sample from which the light is scattered. This filament is perpendicular to the entrance slit of the monochromator. To obtain the maximum signal the image of the filament is rotated by a dove prism so that the image is oriented parallel to the entrance slit. Since the image should completely fill the entrance slit, the optimum sample width is dependent upon the aperture of the collection optics.

The slit height and aperture of the monochromator are 20 mm and f/8.6, respectively. If the aperture of the collection lens is f/1, that lens will produce a magnified



FIG. 1. A cut-away view of the stress apparatus: A—sample; B—upper cup; C—lower cup; D—piston; E—cylinder; F—slots; G,G'—pullrods-H—cap; I—junction insert; J—cross bar; K—set screw; L—pushtube; M—holes in pushtube; N—socket head screws; O—stem; P—interconnecting screw; Q, Q'—O-rings; R—sleeve; S—thumbscrews; T—nylon set screw; U—split nut; V—force transducer housing; W—Amphenol connector; X—channel; Y—outer bellows; Z—inner bellows; AA sleeve; BB—guide rails; CC—nuts; DD—brass ball; EE—hat; FF—ring; GG—force transducer; HH—adapters; II—brass ball; JJ—Swagelock quick-connect.

Rev. Sci. Instrum., Vol 45 No 12 December 1974

focused image of the sample onto the slit of the desired beam aperture of f/8.6 if the magnification is 8.6/1=8.6. Therefore, a sample width of 20/8.6=2.3 mm is desired. In order to obtain a stress of 20 kilobar with a 1.9×2.3 mm sample cross section, a force of 8.92×10^8 dynes must be applied.

The pushtube is under a compressive force and must be designed so as to be stable against buckling. The buckling problem has been mathematically formulated and solved in the Appendix. The solution places constraints on the permissible values of the inner and outer diameters of the pushtube. Another constraint on the outer diameter is that it must be less than the 2.54 cm diam of the sample space of the cryostat that houses the stress apparatus. Also, the presence of the stress apparatus must not affect the ability of an auxiliary pumping system to remove helium gas from the sample space and thereby achieve sub- λ -point temperatures. The cross-sectional area between the pushtube (L) and the sample space wall must be larger than the smallest cross-sectional area already present in the auxiliary pump-cryostat system: that is, 3.54 cm². At the same time, the inner diameter of the pushtube (L) must be sufficiently large so as to permit the passage of the 0.47 cm diam pullrod and wires for electrical connections. For our system, a pushtube with a 2.03 cm o.d. and 1.27 cm i.d. meets the above mentioned criteria and is calculated to withstand buckling forces up to 9.72×10^8 dynes.

The pushtube (L) connects to the stem (O) by an interconnecting hollow screw (P). The stem (O) and the bottom of the force transducer housing (V) are hard soldered together in order to make the junction as strong as possible. The bottom part of the stem is fitted with an O-ring (Q) and fits tightly into the sleeve (R) which is also fitted with an O-ring (Q') and seats in the top of the cryostat. Four thumbscrews (S) in the arms of the sleeve (R) allow fine adjustments in the lateral position of the sample in the cryostat tail piece. These screws sit against the top of the cryostat. The stem (O) is fixed in position by a nylon set screw (T) in the sleeve (R). Fine adjustment of the height of the sample within the cryostat is achieved by turning a split-nut (U) on the threaded part of the stem (O). The split nut (U) rests against the sleeve (R) when the stress



FIG. 2. An isometric view of the sample housing with designations as in Fig. 1.