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Applications of Diamond-Like Carbon Thin Films

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Applications of diamond-like carbon thin films

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There is considerable interest in growing diamond layers and thin films for a wide range of applications. Some of these thin film requirements can be met already using existing diamond-like carbon coatings. This paper reviews the use of diamond-like carbon films in infra red optical, mechanical, electronic and biomedical applications.

1. Introduction

Carbon can exist in many forms, amorphous, glassy and crystalline. It is well known that at ordinary temperatures and pressures graphite is the thermodynamically stable crystalline form of carbon. Diamond can be formed at pressures of 30 kbar† and although it is metastable at atmospheric pressures it reverts to graphite only at elevated temperatures.

The diamond form has quite remarkable physical properties. It is exceedingly hard, has a high Young's modulus and tensile strength combined with high thermal conductivity and thermal shock properties (see table 1). It is chemically inert and in pure form has excellent infra red transmission. There is considerable interest in growing this material at low temperatures and pressures, either as a bulk material, or else as a protective coating on existing materials.

At present the optimum temperature for growing chemical vapour deposition (CVD) diamond is around 900 °C and although this may be lowered to around 600 °C it is still too high for many substrates. In those applications where thin protective layers are adequate then diamond-like carbon (DLC) may provide an immediate solution.

2. Preparation of DLC films

Diamond-like carbon does not have a unique composition but consists of a mixture of amorphous and crystalline phases. Its properties vary considerably with deposition conditions. There were a number of early reports of hard carbon films but most of the current interest was stimulated by the work of Aisenberg & Chabot (1971). They used a carbon ion beam source and accelerated carbon ions towards the substrate by means of a negative bias. They were able to produce films exhibiting many of the properties of diamond and called them diamond-like.

The preparation of similar films from a hydrocarbon gas in a DC plasma was reported by Whitmell & Williamson (1976) and in an RF plasma by Holland & Ohja

† 1 bar = 10^5 Pa.

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Table 1. *Physical properties of diamond*

hardness	10 mg mm^{-2}
Young's modulus	945 GN m^{-2}
thermal conductivity	20 W cm^{-1} K^{-1}
refractive index	2.42
bandgap	5.45 eV

(1976). Since then DLC films have been produced by a variety of techniques including a dual-beam method (Weissmantel 1977), ion beam plating (Bewilogna *et al.* 1979), a simultaneous sputtering and RF plasma CVD process (Green & Lettington 1980; Zelez 1983) and by magnetron sputtering (Savvides & Window 1985).

The essential feature of all of these processes is that the films are grown under ion bombardment and Weissmantel *et al.* (1980) have proposed to call these films i-C for this reason. Many of the deposited layers contain significant amounts of hydrogen (see Angus *et al.* 1980, and enclosed references) and it has been proposed (Bubenzer *et al.* 1983) that these particular materials are called hydrogenated a-C or a-C:H, where a-C stands for amorphous carbon by analogy with amorphous silicon (a–Si). I propose to retain the original name (DLC) introduced by Aisenberg & Chabot (1971).

Diamond-like carbon films contain carbon atoms in a variety of different coordinations. There are the tetragonally coordinated sp^3 carbon atoms present in pure diamond as well as the trigonal sp^2 coordination as found in graphite and possibly some sp^1 coordinated atoms. DLC films may contain microcrystalline diamond and graphite as well as a disordered structure containing a mixture of configurations.

The physical properties of these films depends on the method of preparation. For a given method, the ratio of sp^3 to sp^2 coordinated carbon can be affected by the presence of hydrogen in the films. This ratio has been studied by NMR and XPS (Jansen *et al.* 1985; Grill *et al.* 1987, 1988) as well as IR spectroscopy of hydrogenated material (Dischler *et al.* 1983; Nadler *et al.* 1984). The results differ widely with method of deposition. In the case of the IR spectroscopy measurements, it is not possible to study sp^3 bonded material not containing hydrogen. Also it is difficult to identify diamond crystallites in DLC by Raman scattering but a number of workers have reported the presence of diamond from X-ray scattering measurements. Recently C. T. Pillinger, R. D. Ash, S. A. Russel and J. W. Arden (personal communication) treated a sample of DLC prepared by RF discharge with perchloric acid and obtained at least a 90% weight loss. The residue was studied by an SEM and found to contain a substantial number of individual well-formed octahedral crystals of diamond.

The metastable form of DLC may arise from the energy of the incident ions causing thermal and pressure spikes which are quenched in the depositing layer (see Grill (1988) for enclosed references).

3. Physical properties of DLC films

DLC films can be exceedingly hard. Values in excess of 3000 kg mm^{-2} have been reported (Weissmantel *et al.* 1979; Angus *et al.* 1986). The materials generally have a low coefficient of friction increasing with relative humidity (0.01–0.19) (Enke *et al.* 1980).

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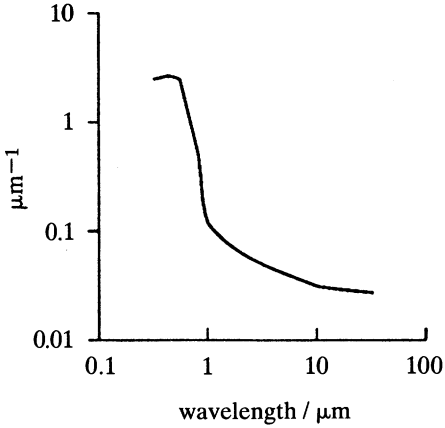

Figure 1. Absorption of DLC.

Layers prepared at high bias potentials have a high internal stress and thicknesses can be limited to 2–3 μm. Such films have been known to shatter germanium substrates and bow optical components.

DLC films are generally absorbing in the visible (see figure 1) but have good transmission in the infra-red region of the spectra and can be used for infra red antireflection coatings on germanium optics (Holland & Ohja 1979; Green & Lettington 1980; Bubenzer *et al.* 1983) and on silicon solar cells (Moravec & Lee 1982).

The coatings can have high electrical resistivity and are chemically durable and abrasion resistant.

4. Diamond-like carbon anti-reflection coatings on germanium

Initially 25 mm diameter test samples of germanium were coated and subjected to laboratory tests. The process was then scaled up to larger components which were subjected to various field trials. Finally the process was used to coat windows and lenses for project applications.

The laboratory testing was to RSRE Specification TS 1888 (Lettington 1985) and required the development of a special abrasion tester. The landbased trials involved a simulated windscreen wiper mounted on the outside of a vehicle undergoing extensive driving trials. The coating showed no sign of deterioration.

For sea trials two of our coated discs of germanium were mounted at sea level on a fort in the Solent along with an uncoated square of germanium. They remained at sea continuously for over four months. The coated samples were virtually unaffected while the uncoated sample was badly corroded and etched away.

The effect of rain impact damage on coated germanium samples was carried out at RAE Farnborough. They were exposed to their standard rain conditions for a few minutes at normal incidence to the rain (2.5 cm dm^{-3}) (Tattershall & Minter 1991) and at 87 m s^{-1} impact velocity. Pitting did occur but the extent was considerably reduced compared to uncoated germanium. The effect of rain damage can be reduced still further through inclining the window to the direction of flight. Our coated germanium samples have also been subjected to single liquid drop impact testing at Cambridge University (van der Zwagg & Field 1983).

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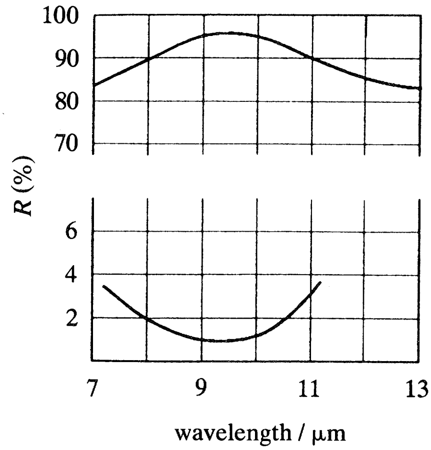

Figure 2. DLC coating on germanium.

A typical optical performance of the now commercially available DLC coating on germanium is illustrated in figure 2. It shows the transmittance of a 1 mm thick germanium substrate coated on the front surface with DLC to OCLI specification 6040011 and with a high efficiency AR coating 6040008, on the rear surface. There is a 5% loss in transmission at the peak wavelength due partly to an index mismatch and partly to absorption in the coating.

5. DLC coatings on zinc sulphide windows

Another of our coating requirements was to protect zinc sulphide infra red windows from rain impact damage. DLC coatings on their own were not adequate since the stress and absorption in the coating limited the thickness that could be used.

This problem was relieved to a certain extent through our development of GeC (Lettington *et al.* 1987). It is a tough durable coating material, with a refractive index a function of composition, that can be designed into multilayer structures. It bonds well to DLC, which is frequently used as the outermost layer. In this form it can act as a stress relieving coating for zinc sulphide windows. These coatings easily pass the RSRE sand/water wiper test of TS 1888.

6. Application of carbon coatings to front surface aluminium mirrors

In current thermal imaging systems, front surface mirrors produced by single point diamond machining of bulk aluminium are used for rotating polygons, flapping mirrors and for relay elements with optical power. The optical performance of these components tends to deteriorate with time and with exposure to the atmosphere. This process can be prevented through the use of a suitable optical coating. Unfortunately the reflectivity of these coated surfaces can be low when used at oblique incidence. This effect has been demonstrated (Cox *et al.* 1975; Lettington & Ball 1981) in aluminium mirrors protected with thin overcoatings of SiO_x and intended for use in the 8–12 μm spectral band. This effect occurs for only one direction of polarization, R_p, parallel to the plane of incidence. Similar effects are

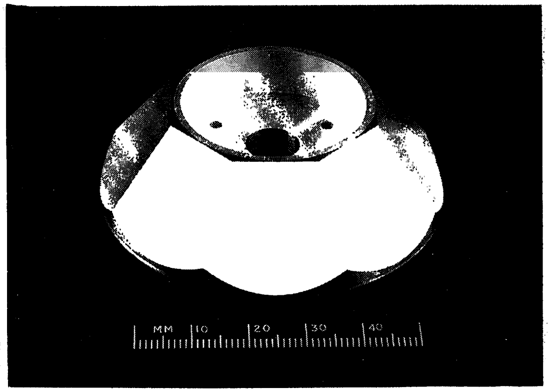

Figure 3. RSRE coated polygon for a coaxial scanner.

observed for many other protective coatings and other metallic reflectors (Pellicori 1978; Cox & Hass 1978) making these coatings unsuitable for use in 8–12 μm thermal imaging systems on 45° mirrors or scanning polygons.

The origin of this effect has been identified and it has been demonstrated that it does not occur in DLC protective coatings (Lettington & Ball 1981). The problem with coatings such as SiO_x is that they have strong optical absorption lines in the spectral region of interest. These coatings are sufficiently thin for this to produce negligible absorption. It does, however, affect the values of n and k in the absorbing region such that the Brewster angle at the air-coating interface occurs at very low angles of incidence. There is destructive interference between this reflection and that at the coating-metal boundary resulting in a loss of reflectivity. This effect does not occur with diamond-like carbon coatings on these mirror surfaces. An RSRE coated polygon for a coaxial scanner is illustrated in figure 3.

7. The use of DLC coatings for photothermal conversion of solar energy

The main aim of photothermal solar energy conversion is to collect solar radiation and to convert it into useful heat. There are two main types of converter: the flat plate collector, where an area of an absorbing material is placed so as to collect the solar radiation; and the focusing collector, where solar radiation is condensed on to a smaller absorbing area. Heat losses from the flat plate collector are high and in the U.K. the operating temperature is rarely higher than 70 °C. The focusing system has higher thermal efficiencies and operating temperatures of several hundred degrees celsius are possible. It is usual to remove the heat from both systems with circulating water.

For either collection process to be effective there must be maximum absorption α of the solar radiation and minimum heat losses. With regard to heat losses, convection and conduction are under the control of the system designer and must be minimized for maximum efficiency. It is the radiation loss ϵ from the absorber which is of consideration here. To maximize the ratio α/ϵ the surface must have high absorption (low reflectivity) from 0.3 μm to about 1.7 μm and low emission (high reflectivity) above 2 μm with a sharp transition between these two regions.

Several spectrally selective coatings have already been proposed and made using silicon or germanium layers deposited on to polished metal substrates (Hahn &

Seraphin 1978; Drummeter & Hass 1964; Seraphin 1979; Janai *et al.* 1979). The use of DLC for this purpose has been proposed by Ball & Lettington (1983). The absorption coefficient of DLC coatings was measured in the visible and infra red regions (see figure 1) and from this and the measured refractive index of about 2.2 values of α, ϵ and α/ϵ were calculated for C, Si and Ge single layer coatings of varying thickness deposited on to aluminium. A single layer of carbon has the highest efficiency. This value, however, it not high enough for most applications and we have sought ways to improve the α/ϵ ratio in multilayer designs.

8. Mechanical applications of DLC layers

In addition to the desirable infrared properties of DLC, discussed previously, the material is also hard and chemically durable making it useful for protecting metal objects from scratching and chemical attack. A variety of metal objects ranging from large sheets to nails, twist drills and machine tool inserts have been coated with DLC. Some of these have remained in the open exposed to the atmosphere for the past seven years without any sign of deterioration. A coated machine tool insert used for cutting aluminium at high speed lasted longer than uncoated inserts but the commercial viability of this coating process was not clearly established.

Other interesting features of DLC are the fact that it is hydrophobic and has a low coefficient of friction. We have coated a number of moving parts inside automobile engines and have successfully reduced wear rates. The frictional properties of DLC have been studied by Enke *et al.* (1980) using a ball-on-disc apparatus. They observed an increase in the coefficient of friction μ from 0.01 to 0.19 with increasing humidity. A sharp increase in μ occurs for relative humidities in excess of 1%. These results are contrary to those for graphite and diamond reported by Bowden & Young (1951) who observed a decrease in μ with increasing water vapour pressure. Similar but more detailed measurements have been reported recently by Kim *et al.* (1991) who included the effects of oxygen on the friction and wear of DLC films. They also considered the application of DLC films as protective overcoats on thin-film magnetic recording discs. Similar applications have been discussed by Tsui & Bogy (1987) and by Marchon *et al.* (1991) who correlated their observations with Raman and resistivity measurements. The application of DLC to magnetic and optical recording discs would appear to offer a promising market opportunity.

Another area in which we had success has been in coating thin optical fibres. A carbon coating inhibited the attack of the silica fibre by moisture so that brittle fracture was less likely to occur (see figure 4). These coatings were also able to restore the properties of aged fibres, probably as a result of the initial back sputtering stage removing some of the surface contamination.

9. Electronic device applications of DLC

DLC films have been studied as both active and passive elements in devices. Their use in an alternating current thin film electroluminescent device has been reported by Kim *et al.* (1990). The emission which occurs during breakdown of the DLC layer is very broad band, extending well into the UV and appears white. However, the brightness and efficiency of current devices are very low.

In another device application Kapoor *et al.* (1986) investigated the use of DLC films as the insulator in metal-insulator-semiconductor (MIS) devices. The results were not

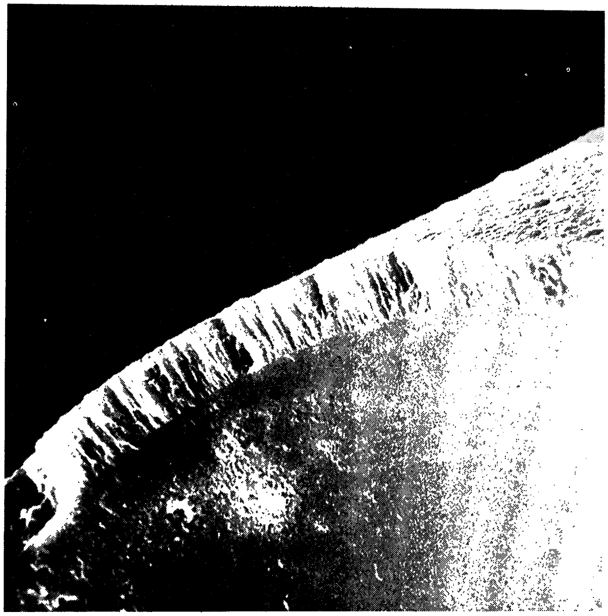

Figure 4. Cross-section of a coated optical fibre.

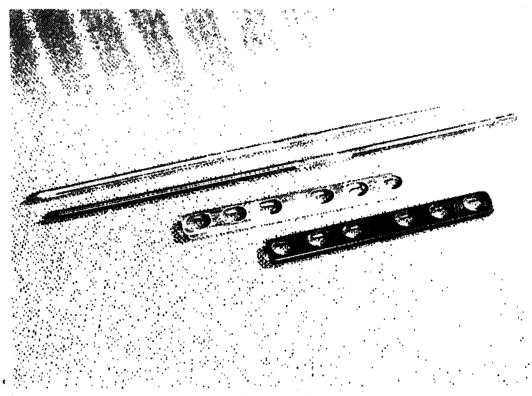

Figure 5. Carbon coated orthopaedic pins.

very encouraging owing to the low resistivity of the films and the large number of defects and traps present either in the layer or at its interface.

In a passive application Rothschild *et al.* (1986) have demonstrated the use of DLC film as a resist for high resolution photolithography of semiconductor surfaces. The process used an eximer laser to induce microchemical etching. A GaAs crystal was covered with a 0.2 μm layer of DLC and exposed to a 193 nm laser pulse. A sharp grating pattern was achieved after using a GaAs wet etchant.

The final device application to be considered uses DLC as an electrical insulator on copper heat sinks for logic and array chips (Marotta *et al.* 1991). The structure consists of an array of copper pistons that are spring loaded into an aluminium heat sink and press down onto the chips, which can dissipate up to 27 W each. The end of the copper piston in contact with the chips was coated first with nickel then

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amorphous hydrogenated silicon and finally with the layer of DLC. This coating prevented scratching of the copper that can occur normally on power on due to a mismatch of expansion coefficients. It also prevented electrical shorts without any deterioration in the thermal conductivity.

10. Medical applications of DLC layers

Carbon in various forms has been used for many biomedical applications during the past two decades (figure 5). Tissue can adhere well to carbon implants and sustain a durable interface. Also in the presence of blood a protein layer is formed which prevents the formation of blood clots at the carbon surface (Jenkins 1980).

Carbon fibre implants can promote the rapid ingrowth of tissue (Jenkins *et al.* 1977) and are used successfully for ligament repair. In bulk form porous charcoal permits a similar ingrowth but has low strength and may also present a site for infection. Carbon can be impregnated for example with resin to improve its properties but the ideal solution is probably to combine the strength of metals with the biocompatibility of carbon in the form of DLC coated metal implants.

Preliminary *in vitro* tests using mouse tissues have shown an encouraging degree of biocompatibility (Thomson *et al.* 1991) as have *in vivo* sheep tests conducted by Professor McGibben of Cardiff Royal Infirmary.

Far more laboratory testing needs to be done before these coatings can be accepted in human trials but if acceptable one could imagine coating many other implants such as the roots of false teeth.

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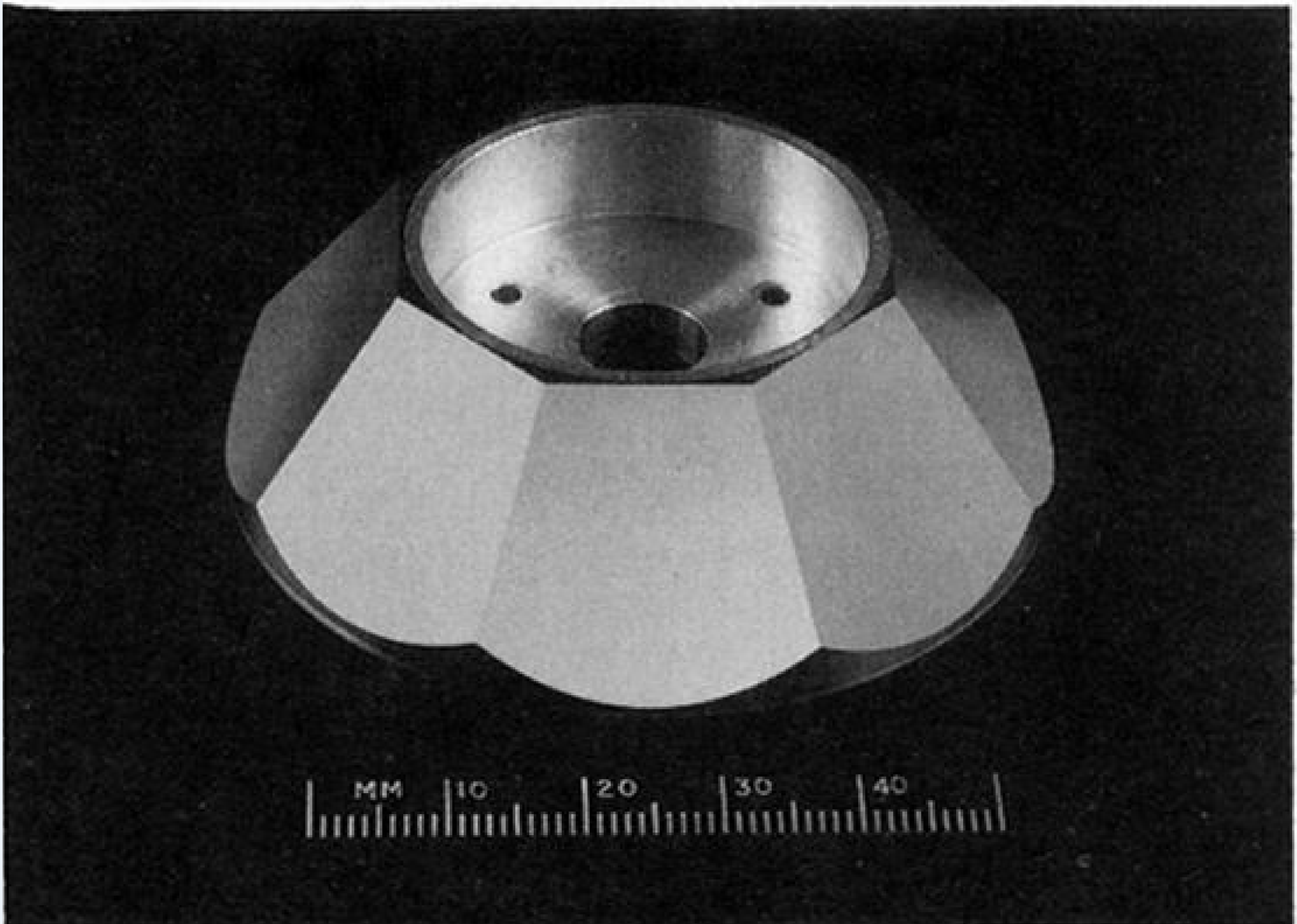

igure 3. RSRE coated polygon for a coaxial scanner.

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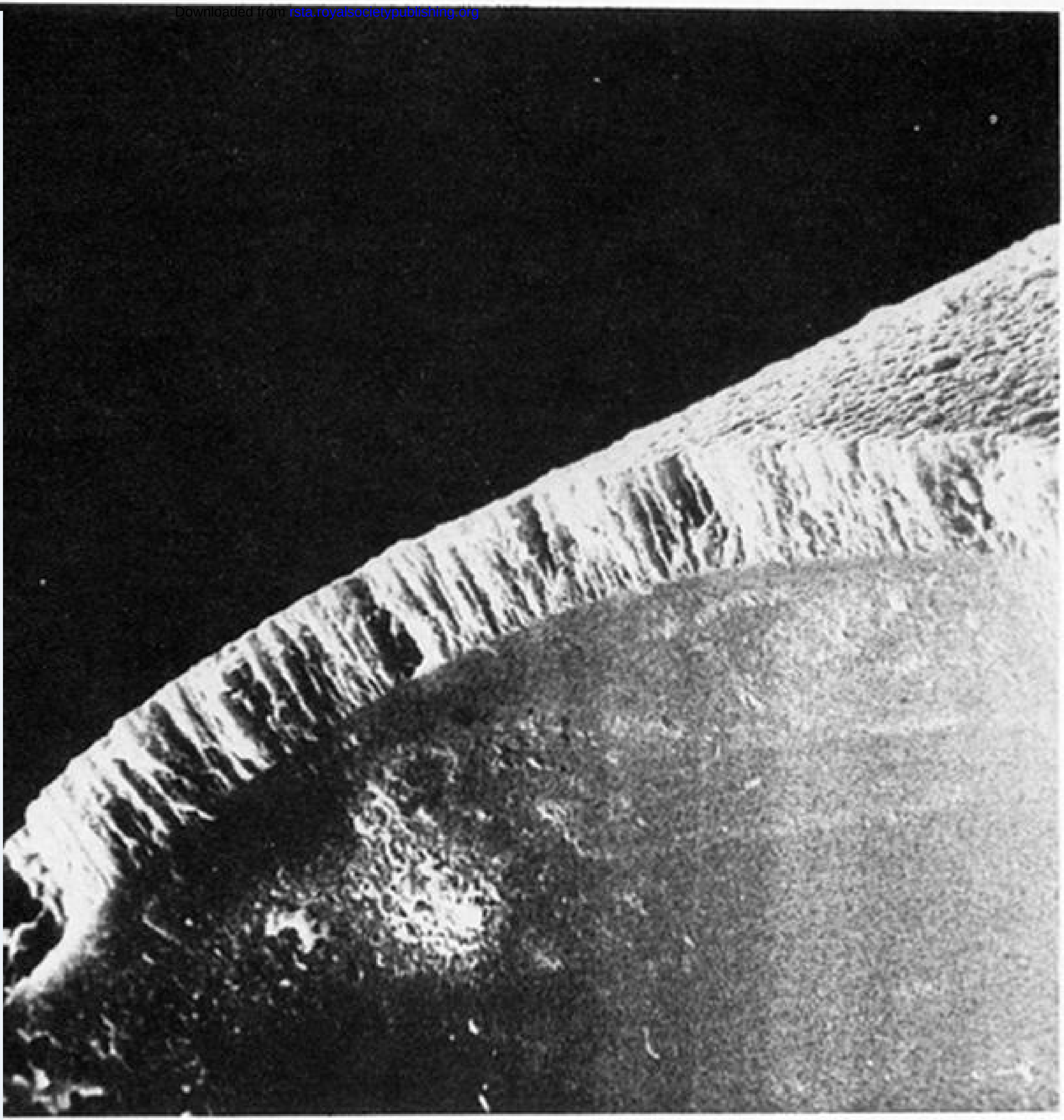

Figure 4. Cross-section of a coated optical fibre.

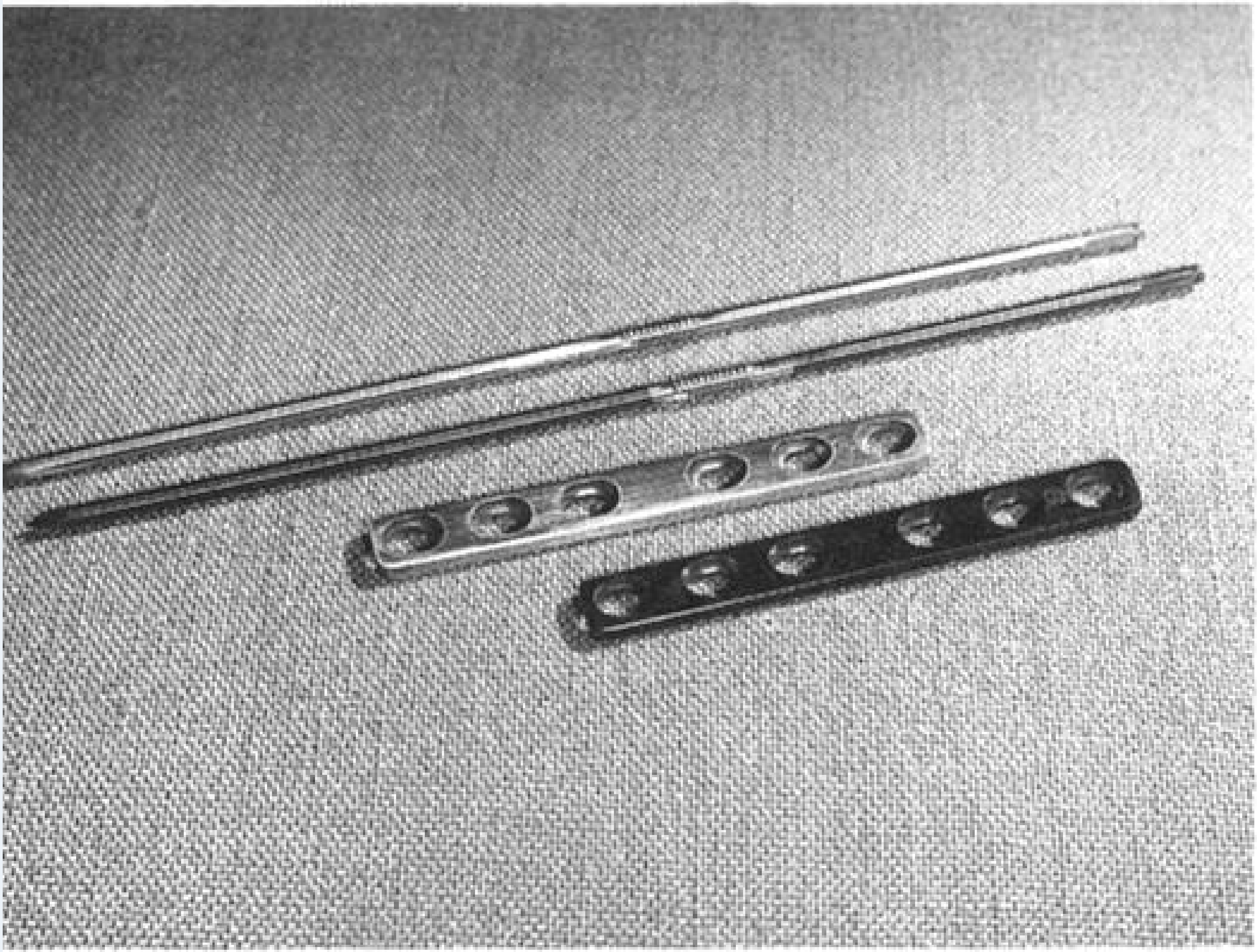

Figure 5. Carbon coated orthopaedic pins.