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Electrically conductive tungsten silicide coatings for EMI/RFI shielding of optically transparent windows

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Conductive metal mesh coatings on external surfaces of infrared (IR) windows reduce the electromagnetic and radio frequency interference (EMI/RFI) but are mechanically soft and easily damaged. Surface-doped semiconductors, such as gallium arsenide, have optical absorption and emission problems, while semiconducting carbide coatings, such as germanium carbide, suffer performance loss at the low end of the required temperature range. Electrically conductive tungsten silicide have been investigated for use in erosion-resistant EMI/RFI protective coatings for IR windows and radomes. Tungsten silicide films were sputtered on zinc sulphide substrates, electrical properties were evaluated, and shielding effectiveness was measured over 400 MHz to 18 GHz. The relevant results are presented and discussed. © *1998 Kluwer Academic Publishers*

1. Introduction

Current infrared (IR) systems are extremely susceptible to electromagnetic interference/radio frequency interference (EMI/RFI) because they possess large apertures that present ideal front-door entry paths for EM radiation. Significant amounts of EM radiation can penetrate and couple to the IR sensor and supporting electronics, which leads to a substantial degradation of system performance. EMI protection is critical to the survivability of the IR system and the system host (e.g. aircraft, satellite). The following are requirements for the EMI/RFI protection on long-wave IR systems:

(i) IR transmission (1.02 and 8–12 μm), ⩾ 90% (less than 10% transmission loss);
(ii) IR angle of incidence: normal ± 15°;
(iii) EMI shielding, ⩾ 30 dB at 400 MHz, ⩾25 dB at 1–4 GHz, ⩾ 20 dB at 5–18 GHz;
(iv) erosion resistance (rain and sand);
(v) low- and high-temperature use;
(vi) strong coating-to-substrate bonding under thermal cycling;
(vii) thermal-shock resistance.

There are two conventional approaches for EMI/RFI protection of IR windows: (1) metal mesh coatings on external structures, and (2) surface-doped semiconductors. Each, however, has its own drawbacks. Metals are mechanically soft and easily damaged by rain and sand erosion. The durability of the metal mesh bond to the window under thermal cycling and thermal shock is also a major concern due to the large difference in the coefficients of thermal expansion (i.e. CTE of ZnS = $7 \times 10^{-6}\,°\mathrm{C}^{-1}$ versus CTE of Al = $25 \times 10^{-6}\,°\mathrm{C}^{-1}$). Surface-doped semiconductors, such as GaAs, suffer from optical absorption and emission problems. Transmission can be reduced by defects, residual porosity (which causes optical scattering), and non-uniformity such as the formation of a gallium-rich phase during fabrication, which can increase absorption [1]. Owing to their reduced electrical conductivity, the EMI/RFI shielding capacity of semiconductors such as silicon carbide and germanium carbide is significantly reduced at low temperatures, while diamond does not have enough electrical conductivity (band gap of 5.4 eV) to be an effective EMI/RFI barrier.

The objective of the present work was to investigate electrically conductive tungsten silicide as an erosion-resistant EMI/RFI protective coating for IR windows to address the problems discussed above. The electrically conductive tungsten silicide coating offers the following benefits:

(i) tungsten silicide coating optimizes IR transmission in the specified wavelength range, in particular

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1.02 μm, and EMI/RFI shielding at the specified frequencies (400 MHz to 18 GHz);

(ii) tungsten silicide coating has a CTE close to that of IR window materials, which reduces debond problems during thermal cycling;

(iii) electrically conductive tungsten silicide is also thermally conductive, which reduces debond problems associated with thermal shock;

(iv) tungsten silicide is expected to shield at both low and high temperatures because its electrical behaviour is similar to that of metals conducting at low temperatures;

(v) tungsten silicide coating is hard and provides erosion protection from sand and rain;

(vi) tungsten silicide film will be stronger than a window made from the same material because the probability of crack initiation and propagation due to material defects is decreased when the volume is minimized.

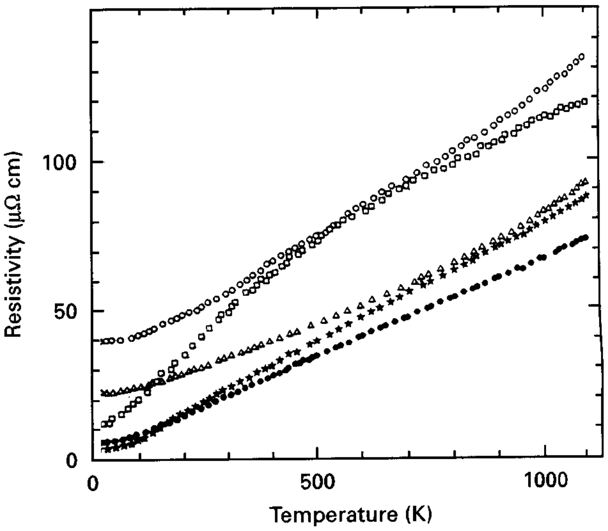

Figure 1 Temperature dependence of the resistivities of some disilicide thin films [2]: (○) $MoSi_2$, (□) $TaSi_2$, (△) WSi_2, (★) $TiSi_2$, (●) $CoSi_2$.

2. Electrical and optical properties of tungsten disilicide

The electrical behaviour of most metal silicides is similar to that of metals – the resistivity, ρ, increases with temperature, as shown in Fig. 1.

For conductors and semiconductors, the key to predicting the optical transmission and EMI shielding is determining the free-carrier contribution to the complex refractive index. The electrical and optical properties of conductors such as metal silicides are controlled by the free-carrier density and carrier mobility. Free carriers cause nearly all of the radio frequency (r.f.) reflection and much of the IR absorption in conductive materials. The shielding effectiveness is reflected in the electrical conductivity of the material, which is controlled by the free-carrier density and carrier mobility. The conductivity, σ, is related to the free-carrier density, N, and the mobility of the carriers, μ, by

$$\sigma = Ne\mu \tag{1}$$

which is related to the sheet resistivity, R_{sq}, by the following relation

$$R_{sq} = \frac{1}{\sigma t} \tag{2}$$

where e is the electronic charge and t is the film thickness. The skin depth, δ (a measure of the attenuation of the electromagnetic radiation in a conductor), and shielding effectiveness, SE, are then related to R_{sq} by the following expressions [3]

$$\delta = \frac{2R_{sq}t}{\omega\mu_m} \tag{3a}$$

and

$$SE \propto \log\frac{A}{R_{sq}} \tag{3b}$$

where ω is the angular frequency of radiation, μ_m is the magnetic permeability of the coating ($\mu_m = 4\pi \times 10^{-7}$ Henry (H) m^{-1} for non-magnetic materials), and A is a constant. The greater the conductivity of the coating, the lower the sheet resistance and the greater the shielding effectiveness. However, if the coating is too conductive, it may absorb the IR and visible radiation that is required to pass through it.

By selecting a high-mobility material, the free-carrier density can be reduced proportionally without sacrificing the desired sheet resistivity. For a given sheet resistivity, the IR transmittance increases with increasing carrier mobility or decreasing free-carrier concentrations. A high mobility also lowers the carrier damping constant, resulting in reduced free-carrier absorption and, thus, improved IR transmission.

Among the metal disilicides, WSi_2 has one of the lowest free-carrier concentrations ($N = 166 \times 10^{20}$ electrons cm^{-3}) and the highest carrier mobility ($\mu = 1.125 \times 10^{-5}\ cm^2\ \mu\Omega^{-1}$ $coulomb^{-1}$) with a relatively low resistivity ($\rho = 12.5\ \mu\Omega\,cm$). Therefore, WSi_2 can potentially have a good IR transmission while concurrently providing a high EMI shielding.

The reflectivity curves for a single-crystal WSi_2 are given in Fig. 2. The reflectivity of WSi_2 in the infrared region falls off very rapidly. The minima of reflectivity with $E//C(R_{\parallel}(\omega))$ and with $E\perp C(R_{\perp}(\omega))$ are located at 0.59 and 0.63 eV, respectively (where E is the electrical field, R is the reflectivity, ω is the frequency, and C is the crystal axis direction). For energies between 0.5 and 3 eV, the values of the absorption coefficient remain rather low, permitting the material to become transparent or partially transparent.

The optical constants, namely, the refractive index, n, and the extinction coefficient, k, of WSi_2 polycrystalline film and bulk single crystals are given in Fig. 3. The values of n and k rapidly decrease from high levels in the FIR region, showing typical metallic behaviour. Near 0.5 eV, the refractive index, n, starts to increase; however, the extinction coefficient, k, shows a plateau with values sufficiently low to permit a significant transmission of incident radiation. For energies higher than 2 eV, k increases and reaches the value of 2.6 at 3 eV, where WSi_2 becomes more and more absorbing

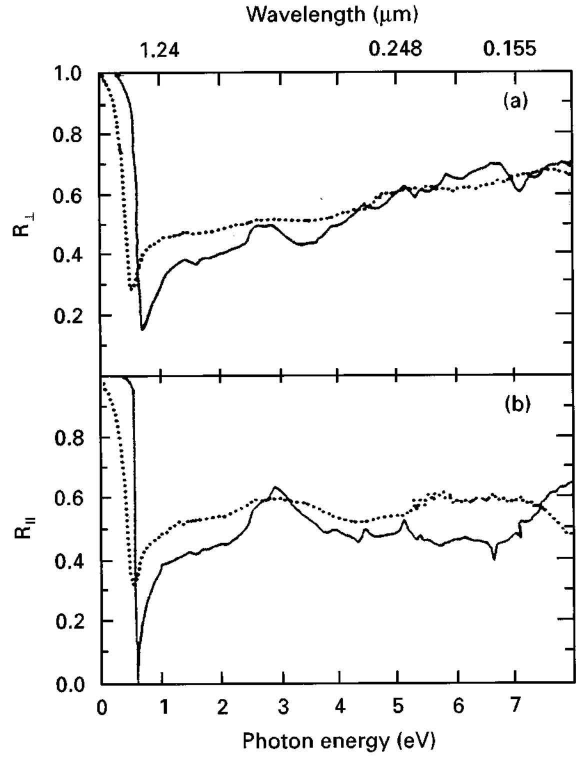

Figure 2 Reflectivity curves of single-crystal WSi_2: (a) field perpendicular to c-axis; (b) field parallel to c-axis [2]. (——) Theory, (– – –) experiment.

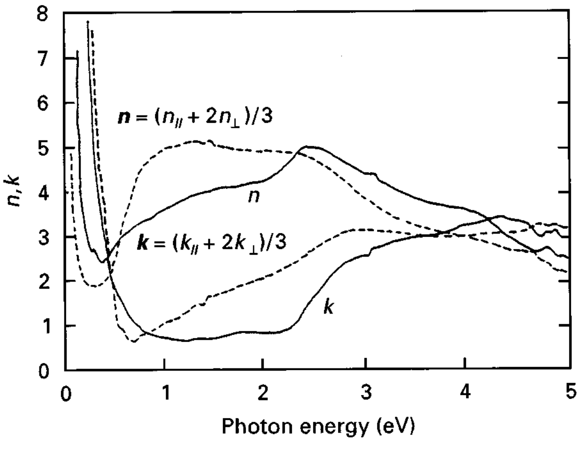

Figure 3 Refractive index, n, and extinction coefficient, k, of (– – –) single-crystal and (——) polycrystalline (bulk) WSi_2 thin films [2].

so that the interference phenomenon disappears. A comparison of n and k for polycrystalline thin films with those for bulk single crystal in Fig. 3 shows a blue shift of the absorption edge and lowering of the n and k values for thin films with respect to those for the single crystal between 0.5 and 2.5 eV.

3. Deposition and properties of WSi_2 thin films

Silicide films from a commercially available WSi_2 target (Cerac, Inc., WI, USA) were r.f. sputtered on polished ZnS substrates. A lack of adhesion was noticed, as evinced by flaking off of the films. Consequently, a thin (< 1.0 nm) titanium film was deposited on the ZnS substrates before silicide deposition to promote the adhesion between the substrates and the thin-film silicide coatings. The use of titanium as an adhesion promoter solved the poor adhesion problem.

Because the sputtered silicide coatings had to be heat treated to increase their electrical conductivities, the chemical stability of ZnS was investigated in an argon environment at 400–900 °C. Annealing at 900 °C for 30 min caused thermal etching of the highly polished ZnS surfaces (Fig. 4a), while annealing at 700 °C and below did not cause any detectable change in the material (Fig. 4b) when compared to the as-polished surface (Fig. 4c).

Since ZnS was thermally etched at temperatures over 700 °C, WSi_2-sputtered ZnS windows were heat treated at 700 °C for 10 and 30 min in an argon atmosphere. The resistivity of WSi_2 films decreased from 97.42 μΩ cm to 14.62 μΩ cm, or in terms of sheet resistivities from $4.33\ \Omega\,\square^{-1}$ to $0.65\ \Omega\,\square^{-1}$, in both cases. The decrease in sheet resistivity with annealing can be explained in terms of microstructural changes due to heat treatment. Upon annealing, the amorphous structure crystallizes into a mixture of disilicides and polysilicides, i.e. $WSi_2 + W_5Si_3$.

Crystallization and grain growth decrease the number of grain boundaries that scatter electrons and increase resistivity. However, the degree of crystallinity and grain size are not the only factors affecting the electrical and optical properties of the silicide films. Silicide films display a range of resistivities. The WSi_2 amount must be optimized in the mixture to obtain the required shielding and IR transmission. The variability is caused by purity, stoichiometry, and the degree of crystallinity. Therefore, the resistivity values reported here may not be optimum and are being further improved upon by controlling the stoichiometry and phase composition to obtain a resistivity lower than 14.62 μΩ cm.

4. EMI shielding measurement

Three 75 mm (3 in.) diameter ZnS windows with 0.7 μm solid WSi_2 thin-film coatings were prepared for testing to verify that the design specifications were met.

The EMI attenuation of the solid films was evaluated. Each window system was tested for EMI shielding from 400 MHz to 18 GHz.

The shielding effectiveness of the metal silicide windows in the required frequency range was tested using a nested reverberation chamber technique [4]. The experimental setup is shown schematically in Fig. 5. In the nested chamber technique, a small reverberation chamber is placed within a large reverberation chamber. The smaller cavity has an aperture containing the window under test. The fields within the nested chamber are recorded with a receiving antenna.

Field homogeneity is critical to testing shielding effectiveness and is guaranteed by a large density of excited modes. The modal density is determined by the quality of the chamber and the bandwidth of the band-limited white Gaussian noise (BLWGN) excitation. The larger the bandwidth, the more modes are

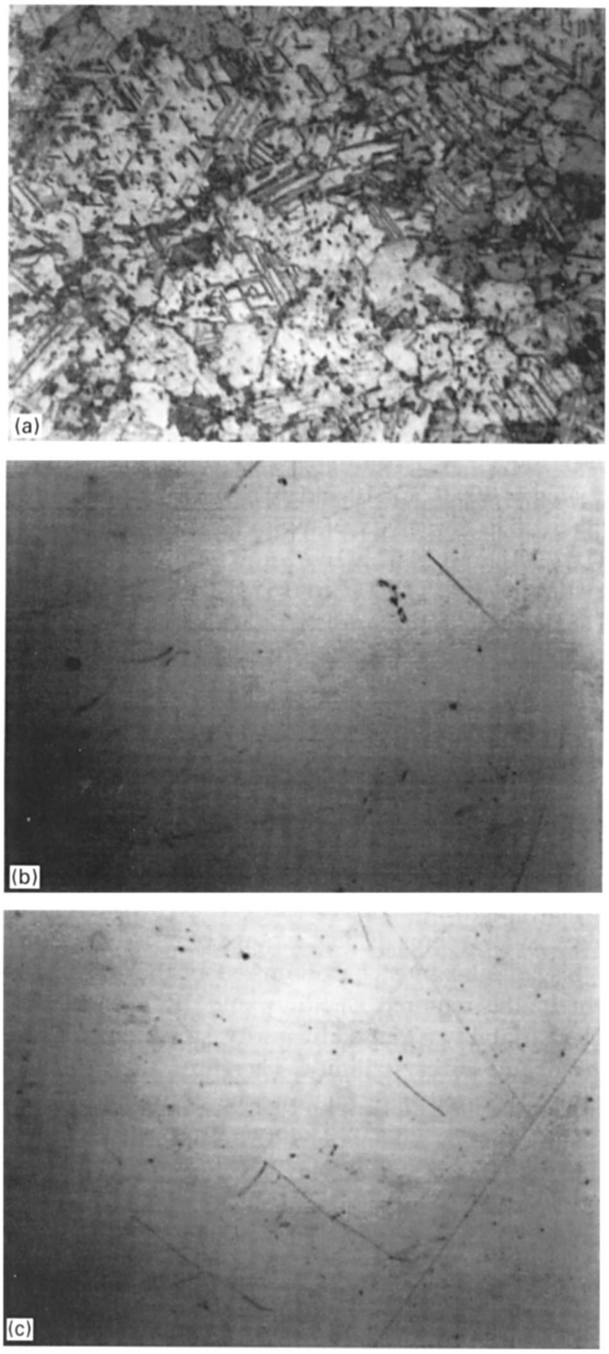

Figure 4 ZnS surfaces (a) after annealing in argon gas at 900 °C for 30 min (note thermal etching), (b) after annealing in argon gas at 700 °C for 30 min, and (c) as-polished.

excited. Field homogeneity was verified spatially with robes in various locations in the chamber and with excitations of various bandwidths.

The shielding effectiveness is a measure of the ability of a material to filter an impinging electromagnetic wave. The shielding ability provided by a given material generally depends on its collective reflection and absorption properties and the phase and polarization of the incoming electromagnetic wave. A shielding factor, ζ, is defined as the ratio of the power density incident on the window, S_1, to the power density transmitted through the window, S_2. The shielding factor is calculated from the following equation [4]

$$10\log\frac{S_1}{S_2} \simeq 10\log\zeta + 10\log\frac{4}{A_{\mathrm{w}}}\left(\frac{2A_{\mathrm{E}}\delta_{\mathrm{E}}\omega}{3c} + \frac{\pi c^2}{2\omega^2}\right) \quad (4)$$

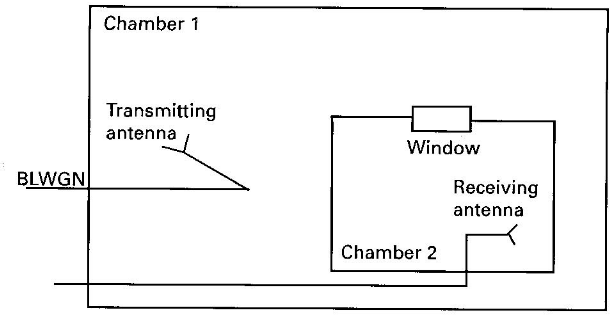

Figure 5 Experimental setup of the nested reverberation chamber technique for electromagnetic shielding measurements. (The directivity of the transmitting antenna is towards the wall to minimize its bias, and the receiving antenna is located inside the smaller, nested reverberation chamber.)

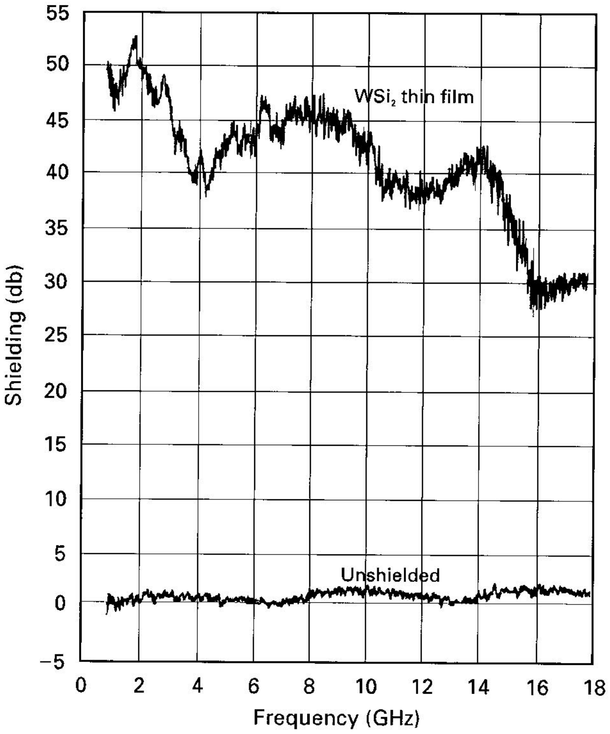

Figure 6 Measured shielding for solid WSi_2 thin-film-coated and unshielded ZnS windows.

where ω, c, A_{W}, A_{E}, and δ_{E} are, respectively, the frequency at which the test is conducted, the speed of light, the window area, the inside surface area of chamber 2, and the skin depth of chamber 2.

In general, determination of the shielding effectiveness using a nested chamber technique requires a correction factor to account for the coupling between the two chambers. However, for this test, the window housing is sufficiently small so that the aperture itself provides sufficient isolation between the two chambers, and the shielding factor can be expressed as the ratio of the power density measured inside the small nested chamber with an open aperture to that of the window.

The results of the test show that the shielding effectiveness of the tungsten disilicide thin film, shown in Fig. 6, exceeds the solicited requirements. A relative measure of the window quality to EMI can be obtained by realizing that the abscissa is the specified requirement in the representative frequency range. Throughout the frequency range from 1–14 GHz, the shielding factor exceeds 40 dB, a factor of 15 dB over the target requirements. In the 1–2 GHz range, approximately 50 dB attenuation is attained. Even the severe drop in the shielding factor at high frequencies results in a measurement of 30 dB, an excess of nearly 10 dB over the specified $\zeta = 20$ dB.

5. Conclusions

1. Tungsten silicide films r.f. sputtered from the WSi_2 target had W_xSi_y multiple stoichiometry and amorphous structure.

2. Sheet resistivities of tungsten silicides decrease with increasing annealing temperatures. However, the annealing temperature and time need to be optimized.

3. The EMI shielding effectiveness of tungsten disilicide solid film exceeds the solicited requirements.

Acknowledgements

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