Si_3N_4 tested in the range 1000 to 1400° C and 17 and 32 for soda-lime glass and alumina, respectively, tested in the presence of water. The referenced papers shown that the variations in the fracture stress σ_f with $\dot{\sigma}$ or $\dot{\epsilon}$ is in the neighbourhood of 10 to $< 20\%$ per decade and that the variations increase with decreasing N and with decreasing average initial flaw size. These analyses also appear to indicate that when the deformation is entirely a result of a process involving cracking as the rate-limiting step in deformation, $N \simeq n + 1$; where n is the exponent relating the crack growth rate, V, to the stress intensity factor K_I

$$V = AK_I^n \tag{4}$$

Lange [9] and Hollenberg *et al.* [10] give expressions of the form

$$\sigma_f = C_1\dot{\epsilon}^{1/(m+1)} = C_2\dot{\sigma}^{1/(n+1)} \tag{5}$$

and since

$$\dot{\sigma} \leqslant \dot{\sigma}_1 = E\dot{\epsilon}_1 < E\dot{\epsilon},$$

where E is the elastic modulus, it is clear that $n > m$. Thus the variation of σ_f with $\dot{\epsilon}$ is greater, although not appreciably greater, than that with $\dot{\sigma}$. This is illustrated in Fig. 1 which contains the data points, without error bars, and the curves of σ_f versus $\dot{\sigma}$ for Si_3N_4 from [9]. To this are added curves and data points for σ_f versus $E\dot{\epsilon}$ obtained from Equation 1 with $N = 10$ which gives $\epsilon/\epsilon_1 = 1.29$. (This limit for $N = \infty$ is $\epsilon/\epsilon_1 = 1.35$.) These curves show only a slight difference in dependence of σ_f on $\dot{\sigma}$ and $\dot{\epsilon}$.

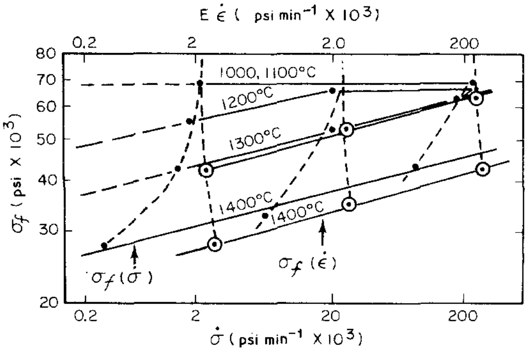

Figure 1 The variation of hot-pressed Si_3N_4 fracture stress with stress rate [9] or strain rate. The dashed curves join points obtained at the same ram speed.

References

1. D. C. LARSON, S. A. BORTZ and R. RUH, Paper 77-WA/GT-4, presented at ASME Annual Meeting, Atlanta, GA, 27 November to 2 December (1977).
2. R. R. WILLS, R. W. STEWART and J. M. WIMMER, *J. Amer. Ceram. Soc.* 60 (1977) 64.
3. D. P. ELIAS and M. W. LINDLEY, *J. Mater. Sci.* 11 (1976) 1278.
4. G. R. TERWILLIGER, *J. Amer. Ceram. Soc.* 57 (1974) 48.
5. H. LARKER, J. ADLERBORN and H. BOHMAN, "Fabrication of Dense Silicon Nitride Parts by Hot Isostatic Pressing", Society of Automotive Engineers International Automotive Engineering Congress and Exposition, Detroit, Mich. 28 February to 4 March (1977).
6. G. G. DEELEY, J. M. HERBERT and N. C. MOORE, *Powder Met.* 8 (1961) 145.
7. W. J. ARRAL, "Ceramics for High Performance Applications", edited by J. J. Burke, A. E. Gorum and R. E. Katz (Brookhill, Chestnut Hill, MA, 1974).
8. J. L. ISKOE, F. F. LANGE and E. S. DIAZ, *J. Mater. Sci.* 11 (1976) 9080.
9. F. F. LANGE, *J. Amer. Ceram. Soc.* 57 (1974) 84.
10. G. W. HOLLENBERG, G. R. TERWILLIGER and R. S. GORDEN, *ibid* 54 (1976) 196.
11. A. G. EVANS, *Int. J. Fracture* 10 (1974) 251.
12. A. G. EVANS and H. JOHNSON, *J. Mater. Sci.* 10 (1975) 214.
13. F. F. LANGE, *Int. J. Fracture* 12 (1976) 739.
14. S. M. WIEDERHORN, A. G. EVANS, E. R. FULLER and H. JOHNSON, *J. Amer. Ceram. Soc.* 57 (1974) 319.

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Preparation and characterization of thin films of FeS_2

Iron pyrite (FeS_2) is a semiconductor with a zero-temperature band gap of about 0.84 eV [1]. In a series of experiments, the various physical properties of FeS_2 have been investigated recently [1]. These studies have been carried out on natural single crystals. To the best of our knowledge, a study on the preparation and characterization of thin films of FeS_2 has yet to appear in the literature. In this letter we report such a study. The films were prepared by evaporation of FeS_2 and they were characterized by the techniques of

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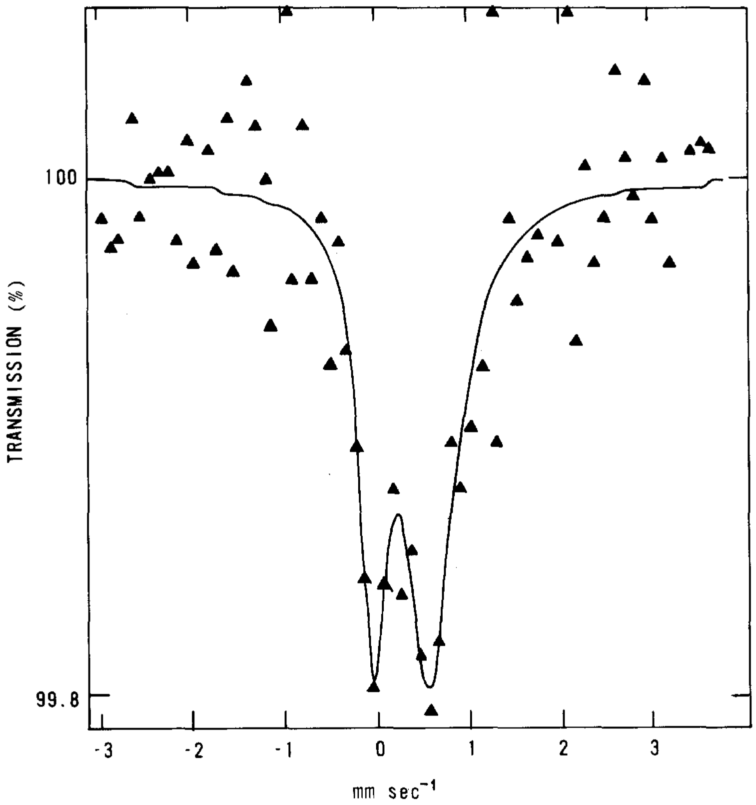

Figure 1 Mössbauer spectrum of the film on Al substrate. The solid line is a computer fit to the experimental points (▲) at room temperature.

Mössbauer and Auger spectroscopies. Film thicknesses up to 0.3 μm were usually obtained.

To prepare thin films by evaporation, a Kinney evaporation unit was used. In one of our earlier attempts, a slow evaporation technique was tried since it is known that in oxygen atmosphere FeS_2 decomposes around 400° C to form other sulphides [2] whereas the melting point of FeS_2 is considerably higher (743° C) [3]. In this experiment, a current of 1 A was passed through the electrodes for a total of 19 h. The pressure in the chamber remained between 2 to 6×10^{-5} Torr and the distance of the glass substrate from the tungsten boat was 6 cm. No significant deposition of FeS_2 took place under these conditions.

The successful experiment in which we believe iron pyrite deposited on glass and Al substrates involved rapid evaporation in the presence of released gases. In this experiment, the starting pressure in the chamber was less than 3×10^{-5} Torr. The current through the tungsten boat was 20 to 24 A for a period of about 5 min. The chamber was isolated from the pumping unit during evaporation in order to deposit FeS_2 in the presence of evolved gases. Consequently the pressure in the chamber rose to 7×10^{-3} Torr at the end of the 5 min period during which evaporation took place. The specimens were left in the chamber overnight (about 15 h) and taken out next morning for analysis.

In Fig. 1, the Mössbauer spectrum of the specimen deposited on Al substrate is shown. The data represents about a 48 h scan and the solid line is the least squares fit to the data using a computer program. Both the centre shift ($\delta = 0.31 \pm 0.03$ mm sec^{-1}) and a quadrupole splitting ($\Delta \simeq 0.62$ mm sec^{-1}) are very close to that measured for a single crystal of iron pyrite [3]. We believe this is important evidence that the material on the film is FeS_2. The films were also examined under a scanning electron microscope. The average particle size is of the order of few μm and a microprobe analysis indicated S : Fe ratio of roughly two although a detailed quantitative analysis was not carried out.

The chemical analysis of the surfaces of the films was carried out using Auger spectroscopy. For comparison purposes, a natural crystal of

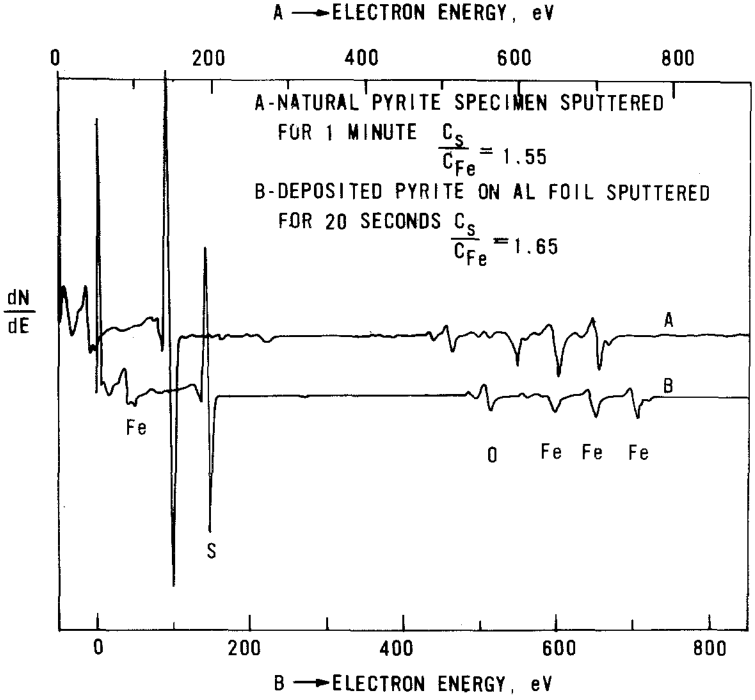

Figure 2 Auger spectrum of natural pyrite (curve A) and the thin film (curve B). The gain for curve A is twice that of curve B. The oxygen peak is believed to be due to contamination.

FeS_2 was also analysed by the same method since quantitative analysis by this technique still has a significant number of uncertainties. In Fig. 2, results of such an analysis by Auger spectroscopy is shown. (Note that the energy scales for the two curves have been shifted relatively in order to distinguish the various peaks). The relative atomic concentration of sulphur (C_S) to that of iron (C_{Fe}) in the thin films is essentially the same as that in a natural crystal. The fact that $C_S/C_{Fe} \neq 2$ even in the natural crystal is indicative of the uncertainties in the quantitative analysis by this method. Note that both specimens were sputtered for short times to remove any surface contaminations before analysis.

In summary, we believe that we have successfully prepared thin films of FeS_2 by evaporation. The experimental evidence described above indicates that the material on the film is in the form of microcrystallites. Since the pressure in the chamber increases significantly during evaporation, some gases are produced during evaporation. The presence of these gases appears to be an important factor in preparing FeS_2. It is quite likely that decomposition of FeS_2 occurs, followed by recombination of Fe and S to form FeS_2. A study of this and other factors affecting the growth of the FeS_2 film is needed.

References

1. W. KOU and M. S. SEEHRA, *Phys. Rev. B* 18 (1978) 7.62.
2. L. A. MARUSAK, P. L. WALKER and L. N. MULAY, *IEEE Trans. Mag.* 12 (1976) 889.
3. R. G. ARNOLD, *Econ. Geol.* 57 (1962) 72.
4. P. A. MONTANO and M. S. SEEHRA, *Solid-State Commun.* 20 (1976) 897.

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