Infrared (8-12 µm) Dome Materials: Current Status

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ABSTRACT

The $8-12~\mu m$ range of infrared radiation being very significant for various electrooptic applications, various materials present themselves as candidates for use as dome (window) materials in this range. This paper discusses various thermal, mechanical and optical properties of these materials. Further, trends in the development of these materials are also presented.

1. INTRODUCTION

Infrared systems find extensive applications in various civilian as well as military areas. These systems are generally classified as either (i) active systems or (ii) passive systems. The active systems involve illumination of the target with an IR radiation of a suitable wavelength and then receiving and analysing the reflected beam. The passive systems operate on the basis of the principle that every object emits infrared radiation at a temperature above zero K, the peak wavelength λ_{max} being determined by the temperature of the body. In either system, the reflected or the emitted radiation is collected on a suitable IR detector, after passing through the optics of the system which collimates it with minimum transmission loss. The collimating system includes various components, such as lenses, prisms, filters, protecting windows and domes.

Various materials have been utilised for the fabrication of these IR transmitting components. These materials differ widely in their transmission ranges, mechanical and thermal properties and stability. A good account of the relationships between the transmission behaviour of a material

and its various structural and mechanical properties has been given by Donald and McMillan¹. Depending upon the application, materials with a certain predetermined set of properties are chosen for the fabrication of the component. Thus, whereas in certain IR laser applications, materials showing high transmission values are required, in the case of IR dome fabrication, materials with high transmission as well as sturdy thermal and mechanical properties are desirable. Refractive index of the material also plays an important role in determining its suitablity for a particular application. Materials with low refractive index can be used for narrow magnification and narrow field of view, but for a wider field of view, materials with high refractive index are required. High refractive index leads to high reflection losses, but this problem can be overcome with the use of anti-reflection coatings.

Most of the IR systems make use of the fact that atmosphere shows three IR transmission ranges, viz., (i) 1-2.4 μ m (ii) 3-5 μ m, and (iii) 8-12 μ m. Most of the optical glasses transmit in 0.1-2.5 μ m range. However, for the 3-5 μ m and 8-12 μ m ranges, special materials are to be selected.

These two ranges are of significant military importance, as these are the IR ranges in which bodies at several hundred degrees and at a temperature in the vicinity of 30 °C emit, respectively. Though, a wide range of materials, including ceramics and glasses are available for fabrication of windows in 3-5 µm range¹, relatively few materials are available for fabrication of domes/windows in 8-12 µm range, which can be used in missile-homing seekers and other airborne IR optical systems. Even, these materials show limitations while considering the future aircraft and supersonic missiles flying at a speed of > 3 Mach. The materials used in fabrication of domes/windows in 8-12 µm range are required to have good transmission behaviour (improved further by the application of antireflection coatings) coupled with good thermal, mechanical, thermomechanical, thermooptical and electrical properties. This combination of properties puts a severe restriction on the choice of materials available for the fabrication of domes/windows in 8-12 µm range.

Currently, ZnS ZnSe, Ge and GaAs are the materials which are being utilised in this region. Calcium lanthanum sulphide (CaLa₂S₄) is being investigated as a potential candidate. Diamond, however, remains the most attractive material because of its excellent properties. Diamond can be used either as an antireflection/protective coating on the conventional IR transmitting materials, e.g. ZnS or itself as a free standing IR transmitting window. Efforts are on to explore the possibility of using diamond (type-II) by making it available in thicker layer or bulk form. This article reviews the methods of preparation of these materials as well as their present status.

2. METHODS OF SYNTHESIS

2.1 Zinc Sulphide

Zinc Sulphide (ZnS) is the most suitable among the present generation of materials for transmission in 8-12 μ m range under conditions where temperatures of several hundred degree centigrade are to be encountered. Though single crystals of

ZnS have been grown, their use in IR window fabrication has been restricted owing to the high melting point (accompanied by dissociation) of ZnS leading to the complexities of the process involved. Moreover, fabrication of a suitably-shaped body from a single crystal proves to be a tedious process. As a result, polycrystalline ZnS body is utilised in the fabrication of IR window. Polycrystalline ZnS body can be produced in two ways: (i) synthesis of ZnS powder through different conventional chemical reactions followed by its compaction using hot pressing or hot isostatic pressing (HIP). and (ii) chemical vapour deposition (CVD) of ZnS on a suitably-shaped substrate through a vapour phase reaction. The body formed can be further strengthened/compacted by an HIP technique.

2.1.1 Conventional Chemical Reaction Routes

ZnS powder has been prepared through different chemical routes. Notable among these are: (i) aqueous chemical reactions involving a soluble Zn salt and H_2S , thiourea or thioacetamide and (ii) vapour phase reaction between elemental Zn and S in a closed ampoule. Of these, the former have been investigated thoroughly, even though the literature on both these routes is scanty.

A number of workers have studied the $ZnSO_4$ (aq)- H_2S reaction. Two subprocesses have been developed as a result: (i) acid process, and (ii) alkali process. These two processes differ in the pH conditions of the reactions. The reactions involved in the two processes are:

(a) Acid Process

$$ZnO + H_2SO_4 \rightarrow ZnSO_4 + H_2O$$

 $ZnSO_4 + H_2S \rightarrow ZnS + H_2SO_4$

(b) Alkali Process
$$ZnO + 2HCl \rightarrow ZnCl_2 + H_2O$$

$$ZnCl_2 + 2NH_4OH \rightarrow Zn(OH)_2 + 2NH_4Cl$$

$$2Zn(OH)_2 + 4NH_4OH + 4NH_4Cl \rightarrow 2Zn(NH_3)_4Cl_2$$

$$+ 8H_2O$$

$$Zn(NH_3)_4Cl_2 + H_2S + 2H_2O \rightarrow ZnS + 2NH_4Cl$$

$$+ 2NH_4OH$$

The acid process is considered to be better suited, as the alkali process is known to introduce undesirable hydroxide species². Eastman Kodak has used acid process. After purification, the *ZnS* is hot pressed to give a transparent body marketed as IRTRAN-2.

ZnS powder³ is produced by the reaction of ZnSO₄ with thiourea also, but the powder is contaminated with undesirable hydroxide impurities and hence has little use in fabrication of IR windows.

Zn salt reaction with thioacetamide has been investigated and uniformly-shaped spherical particles have been formed⁴. The method involves the following reactions:

$$H_3^+O$$

$$CH_3CSNH_2 + H_2O \longrightarrow CH_3CONH_2 + H_2S$$

$$Zn^{2+}(aq) + H_2S + 2H_2O \longrightarrow ZnS + 2H_3^+O$$

These powders have been found to give on compaction near-theoretical density bodies. However, IR transmission behaviours of these compacts have not been discussed⁵. Production of ZnS powder by vapour phase reaction of elemental Zn and S has been studied⁶. However, IR transmission characteristics as well as other physical properties of these powders have not been investigated.

Very little information is available on hot isostatic pressing/(HIP) of ZnS powder. Carnall⁷ of Eastman Kodak studied the effect of hot pressing conditions on the density of compact ZnS. Keizo Uematsu, et al. studied the effect of additives, such as Bi_2S_3 , Al_2S_3 and Li_2S on the grain size and density of hot pressed ZnS. Huffadiene9, et al., discussed the HIP conditions of ZnS powder and concluded that in order to retain the transparency, the HIP sample is to be maintained under pressure while cooling. More recently, Xue and Raj¹⁰ investigated the effect of hot pressing conditions on the optical transmission of ZnS. They reached the conclusion that the relative proportions of two ZnS phases, viz., (i) cubic-sphalarite phase, and (ii) hexagonal-wurtzite phase, as well as the grain size

of the compacted body, play a crucial role in determining the IR transmission of the body.

2.1.2 Chemical Vapour Deposition

Chemical vapour deposition (CVD) technique was evolved as an alternative to meet new requirements of size, purity, durability and economics. Scientists working at Raytheon Co. 11 USA, pioneered the early work in this field.

Simply stated, CVD is a gas phase reaction which results in the deposition of a solid material on a suitably-shaped substrate or mandrel. Thus, Zn vapour and H_2S gas are allowed to react at an elevated temperature (600-800 °C) and reduced pressure (<100 torr) in a reactor. The reaction involved is:

$$600 - 800$$
 °C
 $Zn(v) + H_2S(g)$ \Rightarrow $ZnS(s) + H_2(g)$
 $< 100 \text{ torr}$

The deposition rate can be in the range $10-100 \mu/h$. Thus, a period of almost three weeks is required for growing about 1 in. thick ZnS crust.

To improve upon the physical properties, the CVD-grown material has been subjected to HIP. The material thus obtained has been called multispectral material (showing transmission in both visible and IR regions). Savage, ¹² et al. studied different characteristics of the multispectral material and the role of HIP parameters.

2.2 Zinc Selenide

2.2.1 Chemical Synthesis

Chemical synthesis of zinc selenide (ZnSe) in an aqueous solution is much involved compared to that of ZnS because of the instability of freshly precipitated ZnSe. This problem has been circumvented by dissolving Se in Na_2SO_3 solution and then making this solution react with Zn salt solution ². Taylor¹³, et al, reacted a mixture of Se, activated charcoal, KOH and $KH_2PO_2.2H_2O$ (potassium hypo phosphite) in H_2O with $ZnCO_3$ to produce ZnSe. Korezynski¹⁴, et al. reacted Zn salts with K_2Se under H_2 atmosphere to precipitate ZnSe, which, however, is non-stoichiometric.

ZnSe powder has been prepared by reacting elemental Zn and Se in an evacuated $^{15}SiO_2$ ampoule at 450-750 °C. Hot pressing of the ZnSe powder has been standardised at Eastman Kodak and the resultant product has been marketed by them as IRTRAN-4.

2.2.2 Chemical Vapour Deposition

ZnSe is also produced by CVD technique. This technique is similar to that employed for preparing ZnS. The following reaction takes place in the reactor:

600-800 °C

$$Zn(v) + H_2Se$$
 $ZnSe + H_2$

< 100 torr

2.3 Zinc Sulphide/Zinc Selenium Sandwitch Windows

ZnS is sturdier then ZnSe as far as physical (mechanical/thermal) properties are concerned. However, transmissionwise, ZnSe is superior to ZnS. Hence, ZnS/ZnSe sandwitch windows have been developed involving a CVD-grown ZnSe substrate coated with a thin layer of ZnS to provide mechanical/thermal support ¹⁶.

2.4 Germanium

Germanium (Ge) has been one of the earliest leaders in the field of IR transmitting materials because of its excellent transmission range. Single crystals of Ge used in the fabrication of IR windows are easily grown by Czochralski technique. In this technique, Ge is melted at 900-1000 °C in a large container and a Ge seed of the required orientation is lowered in the melt and then gradually withdrawn at a very slow rate. Bulk crystals of Ge up to 26 cm diameter have been grown.

Polycrystalline Ge can also be grown by the Bridgeman technique in which the melt is gradually moved through a preset temperature gradient.

2.5 Gallium Arsenide

Gallium arsenide (GaAs), another versatile IR transmitting material, can be grown in single crystalline or polycrystalline form. Both Bridgeman and Czochralski techniques have been used. However, these conventional techniques lead to the growth of ingots of limited size only owing to the weak strength of quartz ampoules. Halfner and Braun ¹⁷ made use of He vented ampoules to grow large crystals of GaAs. Kloeck ¹⁸, et al. developed polycrystalline GaAs body by hot pressing GaAs powder (formed by grinding GaAs single crystal) to get over the problem related to its weak fracture toughness.

2.6 Calcium Lanthanum Sulphide

Calcium lanthanum sulphide ($CaLa_2S_4$) is one of the members of the family of ternary sulphides with the general formula AB_2S_4 (where A is a divalent metal cation and B is a trivalent metal cation). This compound has recently been investigated and has been found to be a promising IR transmitting material in the 8-14 μ m range ^{19,20} besides being inexpensive. The material is synthesised in powder form through ceramic route. White, ¹⁹ et al. used $CaCO_3$, $La(OH)_3$ and H_2S as the starting materials. They mixed the first two in stoichiometric ratio and then fired the resulting mixed powder in an atmosphere of H_2S at 1100 °C for 3-7 days.

$$CaCO_3 + 2La(OH)_3 + 4H_2S \rightarrow CaLa_2S_4 + CO_2 + 7H_2O$$

Covino, 20 et al. have developed another technique, wherein a stoichiometric mixture of Ca $(NO_3)_2$ and $La(NO_3)_2$ in HNO_3 is allowed to dehydrate and the resulting fine mixture of nitrates is treated with H_2S gas at about 900 °C to give $CaLa_2S_4$ powder.

The $CaLa_2S_4$ powder obtained through either of the above methods is then hot pressed/cold pressed to give green compact of > 55 per cent of theoretical density, which then is sintered in flowing H_2S gas at 1050-1200 °C to give a ceramic body of > 95 per cent of the theoretical density. This is then isostatically hot pressed 990-1200 °C

and at 172-200 MPa for 15 min to 3 hr to give a transparent body. The most suitable composition range has been found to be between $40CaS.60La_2S_3$ and $50CaS.50La_2S_3$.

2.7 Diamond

Diamond is the material considered to be the ultimate choice for window applications in the 8-12 µm range because of its excellent optical, thermal and mechanical characteristics. IR grade material diamond (type-II) was used in space optics as a window material in the pioneer Venus IR radiometer ²¹.

Until early eighties, the synthesis of diamond was carried out using a high pressure-high temperature (HP-HT) process which led to the formation of diamond particles used mainly in grinding application²². However, in 1983, Kamo,²³ diamond in a microwave et al. synthesised plasma-assisted CVD process. Since then, a number of papers dealing with the growth of diamonds in a microwave plasma-assisted CVD process have appeared. Kobashi, 24 et al. reviewed this work up to 1987 (including the earlier processing work done by the Russians in the field of diamond synthesis using chemical transport reaction (CTR) method, gaseous phase decomposition, etc.) and presented an exhaustive account of this work.

Setaka, 23,25 et al. investigated three types of CVD methods for diamond synthesis: (i) hot filament method, (ii) rf-plasma CVD method, and (iii) microwave plasma CVD method. In the hot filament CVD setup, a W filament placed about 10 mm above the substrate is electrically heated to about 2000 °C. The quartz tube reaction chamber is placed in a furnace at ~ 600 °C. Because of hot filament radiation and furnace radiation, the substrate gets heated to about 1000 °C and various surface processes, e.g. molecular dissociation, diffusion, chemical reactions, etc. get activated. The hot filament creates atomic H and various active fragmantary hydrocarbon species, (ions, radicals) by dissociating molecular H_2 and CH_4 . These active species having long life times, travel to the substrate surface causing growth of diamond.

In rf-plasma CVD setup, a hydrocarbonhydrogen mixed gas plasma is generated, by way of irradiation by rf-plasma wave, and as a result diamond grows on the substrate.

Microwave plasma CVD setup is similar to the rf-plasma setup, except that plasma is generated by microwaves. The mechanism of diamond formation in microwave plasma CVD, which is currently receiving maximum attention is not fully understood. According to a mechanism for diamond synthesis proposed by Setaka²⁶, CH₄ and H₂ are dissociated in plasma and chemically active fragmantary hydrocarbon ions, radicals and atomic H are generated. The former migrate to the substrate and are adsorbed. Initially, C clusters are formed that have predominantly graphitic and amorphous structures because of thermodynamic considerations. However, metastable diamond structures are also formed. Since graphitehydrogen reaction is very fast, graphite-cluster structures are quickly removed, whereas diamond structures stay and grow.

Whereas CH_4 - H_2 mixture has been employed in thermal and hot filament CVD techniques, other hydrocarbon gases, such as C_2H_2 , C_2H_4 , C_2H_6 , have also been used in microwave plasma CVD techniques.

Recently, Ravi 27 reported high deposition rate synthesis of diamond using a technique involving C_2H_2 - O_2 combustion reaction, which activates excess C_2H_2 in the gas mix, leading to deposition of a diamond film on a temperature-controlled substrate. Harris 28 reported the production of 0.3-1.0 mm thick clear diamond windows having diameters up to 6 cm.

3. PHYSICAL (OPTICAL, THERMAL & MECHANICAL) CHARACTERISTICS

Materials required for fabrication of IR windows in 8-12 μ m range should be mechanically robust, thermal shock resistant and thermally conducting (so as to have minimum heating effect) and of course, be excellent transmitters in this

range. It is needless to say that existing materials do not possess ideal characteristics in all areas of requirement. In fact, some characteristics vary in opposite direction. For example, materials possessing strong bonds between constituent atoms show very good mechanical characteristics, but the transmission range becomes narrow and the cutoff wavelength shifts towards shorter wavelengths. On the contrary, materials having weak inter-atomic bonds (and consequently poor mechanical properties) show a longer transmission range. Thus, a trade-off has to be resorted to while choosing a material for a particular application. However,

though still in the development stage, diamond remains an excellent potential material with excellent all-round characteristics, except for two absorption bands (one at $\sim 5~\mu m$ and the other at $\sim 20~\mu m$) which do not fall in the 8-12 μm range (Table 1). The relative characteristics of these materials are discussed below.

3.1 Optical Properties

Refractive indices and transmission ranges of the various materials discussed above are presented in Table 1. Transmission of 1 mm thick samples of ZnS, ZnSe, GaAs, Ge, 45CaS. 55La₂S₃ and a 2 mm thick type-IIa gem diamond at indicated

Table 1. Optical properties of some IR transmitting materials (8-12 $\mu\text{m})$

Material T	ransmission range	Absorption coefficient					Refractive index	dn/dt
	(μ m)	(cm ⁻¹) at					(10 µm)	(°C ⁻¹
		3.33	5.0	8.3	10	12		
		(µm)	(µm)	(µm)	(µm)	(µm)		
Ge	1.8-15			0.02	0.03	0.2 (25 ° C)	4.0032	27 x 10 ⁻⁵
			0.17	0.58	0.66	0.94 (100 °C)		
GaAs	1.8-16	< 0.01	< 0.01	< 0.01	< 0.01	0.09 (25 ° C)	3.2769	· -
			< 0.01	< 0.08	0.15	0.42 (400 °C)		
ZnS (CVD)	1-12	0.02	0.02	0.08	0,.08	(25 °C)	2.2002	4.33 x 10 ⁻¹ (10 μm)
			0.02	0.15	0.15	(600 °C)		
ZnSe (CVD)	0.6-22	< 0.01	<0.01	< 0.01	< 0.01	(25 °C)	2.4070	6.1 x 10 ⁻⁵ (10 µm)
			<0.01	< 0.01	< 0.01	(600 °C)		
42.5 CaS- 57.5 La ₂ S ₃	2-15			0.2	0.17	0.5	~2.53	
2 3				(App.	Abs. Coeff.)			
Diamond with 2 absorption bands	•	1	12	< 0.03	<0.03	<0.03 (25 °C)	~2.38	
~5 µm and ~20 (synthetic 2 mm thick layer)		2.5	18	< 0.03	< 0.03	< 0.03 (600 °C)		

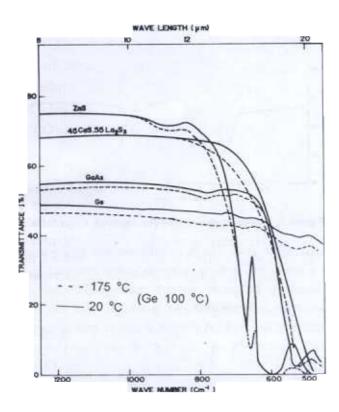


Figure 1. Transmittance of 1 mm thick samples of ZnS, GaAs, Ge and 45 CaS. 55 La₂S₃.

temperatures are shown in Figs 1-3. Though Ge shows good transmission up to ~12 µm with slow decline in the transmission behaviour up to 20 μm , free electron absorption restricts its use only up to 70 °C and hence, it cannot be used in such applications where major aerodynamic heating is involved. GaAs shows good transmission up to 16 µm, but its use is also restricted up to 200 °C. ZnS though suitable at higher temperatures because of its larger energy gap, has got a relatively shorter transmission range with its transmission being useful up to 12 µm. CVD ZnS shows better transmission behaviour than hot pressed samples and the former can be further improved through a post-deposition treatment; the resulting body is known as 'Cleartran' ZnS 16.

Xue and Raj¹⁰ investigated the role of hot pressing conditions on the optical transmission of *ZnS* and concluded that grain size and phase composition play key roles.

 $CaLa_2S_4$ shows slightly lower transmission than ZnS_5 , but its range is relatively longer. ZnSe shows maximum transmission among the existing

Table 2. Mechanical and thermal properties of some IR transmitting materials (8-12 um)

Property	Ge	GaAs	7nS	ZnSe	42.5CaS	Diamono
- roperty	Ue	Guns	CVD		-57.5La ₂ S ₃	Diamon
Density (kgm ⁻³ 10 ³)	5.32	5.32	4.08	5.27	4.26	3.515
Hardness (GPa)	8.3	7.4	2.2	1.0	5.6	88
Young's modulus (GPa)	103	85	75	70	96	1050
Fracture toughness (MNm ^{-3/2})	0.6	3	0.65	0.7	0.53	
2 mm water drop damage threshold velocity (m/s)	205		170	137- 152	250	
Thermal expansion coefficient X 10 ⁻⁶ / °C	6.1	5.7	7.4	7.57	14.7	0.8
R'	8.0	3.7	2.1	1.4	0.7	5604
Thermal conductivity (W/mK)	70	35	17	18	1.7	2000
Poisson ratio	0.279	0.28	0.29	0.28	0.25	~0.2

materials, its range extending up to 18 μm even for thicker windows (thickness~14 mm). Temperature-degradation of transmission is also least in the case of ZnSe. As ZnSe is mechanically weak, ZnS/ZnSe composite windows have been fabricated. A typical transmission curve for (6 mm ZnSe +1 mm ZnS) window is shown in Fig. 2. Transmission behaviour of diamond shows that it is the best material for use in 8-12 μm range.

3.2 Mechanical Properties

Various mechanical properties of some IR transmitting materials are given in Table 2. It is apparent that most of the mechanical properties of the emerging material, $CaLa_2S_4$, are either superior to those of ZnS or approach them. Particularly significant is the threshold velocity for damage by

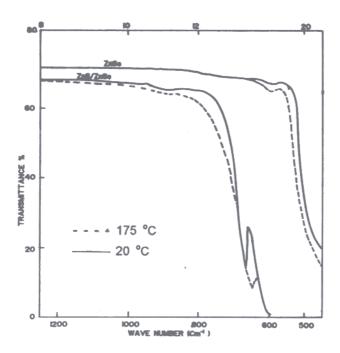


Figure 2. Transmittance of 1 mm thick sample of ZnSe and (1 mm ZnS + 6 mm ZnSe) laminate.

rain drops. Ge and GaAs are mechanically superior to ZnS, but their temperature range is limited, leaving the competition between ZnS and $CaLa_2S_4$. Diamond, of course, is a much superior material when mechanical properties are the criteria.

3.3 Thermal Properties

During high speed flights, the window materials become hot, and their optical properties are likely to be impaired. Ge and GaAs, with low energy gaps, develop the capacity for increased absorption of radiation due to increased free electron generation. ZnS is better and ZnSe is still more useful in this regard. Materials should be thermal shock resistant. A thermal shock figure of merit ²⁹ has been defined as:

$$R' = \sigma k (1-v) / a E$$

where σ is fracture strength; ν , Poisson's ratio; k, thermal conductivity; a, thermal expansion; and E, the Young's modulus. R' values of the materials are listed in Table 2. It is observed that Ge and GaAs have better R' values than ZnS which is superior to ZnSe and $CaLa_2S_4$ 6. The last named is not at all useful for windows which experience a sudden

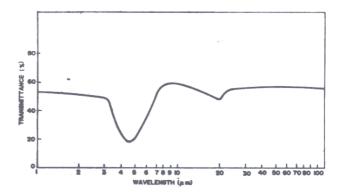


Figure 3. Transmittance of type IIa diamond, 2 mm thick

temperature rise. These considerations again point to a trade-off among various materials and ZnS appears to be most suitable among the existing materials. Diamond, as pointed out earlier, possesses the highest potential and is way ahead of other materials in respect of various thermal properties.

4. CONCLUSION

Electrooptic systems, the platforms they perform on, and their missions, continue to place increasing requirements on IR domes and windows associated with them. Thus, more and more robust, optically transparent and thermally stable materials are required. The materials currently available for these requirements, such as Ge, GaAs, ZnS, ZnSe, emerging materials like CaLa₂S₄ and the most ideal material, diamond, are discussed in the light of these requirements. Methods for their synthesis and their characteristics have been reviewed.

It appears that other than diamond, no other material is suitable for all applications. Up to 100 °C, Ge is quite suitable. For window applications up to 200 °C, GaAs is quite good, whereas for window temperatures higher than 200 °C, there is close competition between ZnS, ZnS/ZnSe laminates and CaLa₂S₄. Depending upon the requirement, some characteristic may be traded-off for some other and choice of the material made accordingly. Diamond is the ideal material for these applications. Recent developments in the field of synthetic diamonds have been discussed.

ACKNOWLEDGEMENT

The authors are thankful to Shri A. Mallik, Director, Defence Science Centre, Delhi, for his keen interest in this work and permission to publish this paper.

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