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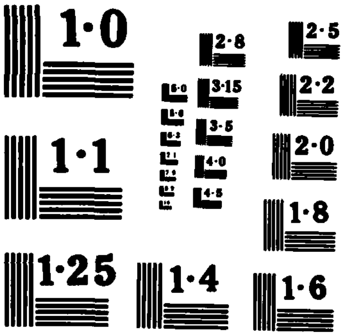

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AIR FORCE SYSTEMS COMMAND

INVESTIGATION ON THE ABILITY OF ANTIREFLECTION COATING TO WITHSTAND THE DESTRUCTIVE EFFECTS OF LASER RADIATION IN IR RANGE (10.6 μm)

by

Wang Jingrui, Liu Jien

EDITED TRANSLATION

FTD-ID(RS)T-0186-85 15 August 1985

MICROFICHE NR: FTD-85-C-000668

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English pages: 12

Source: Dianzixue Tongzun, Vol. 6, Nr. 3, 1984,
pp. 250-254

Country of origin: China
Translated by: FLS, INC.
F33657-85-D-2079
Requester: FTD/TQTD
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INVESTIGATION ON THE ABILITY OF ANTIREFLECTION COATING TO WITHSTAND THE DESTRUCTIVE EFFECTS OF LASER RADIATION IN IR RANGE (10.6 μm)*

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(Institute of Electronics, Academia Sinica)

Micrometers

In this paper the ability of antireflection coating to withstand the destructive effect of laser radiation in the neighborhood of 10 μm is investigated. Some methods for improving the destructive threshold of the antireflection coatings are proposed.

I. Introduction

Antireflection (AR) coating has been widely used in laser systems to raise the auto-vibration threshold of the working material, to increase the output power and to reduce the loss of power due to optical components. Since the laser output power keeps increasing, optical coatings are faced with rising demand for better laser resistance capability. The destructive thresholds of

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*Received on April 27, 1983.

the coatings have also become the main design parameter for coatings which work under a high-current, pulsed high-power CO_2 laser.

From the early 70's, the U.S. National Bureau of Standards founded a specialized publication covering laser induced destruction of various types of optical materials. Dr. B. E. Newnam of the Los Alamos Scientific Laboratory, U.S.A., has done major work in this field, especially in the area of destruction of 10 μm AR coatings [1]. We have also done some research on raising the destructive threshold of the AR coating of the CO_2 laser. We made a rather detailed examination of the AR coatings for Ge substrates. We have analyzed, one by one, the effects of the selection of the substrate and its machining, thin-film manufacturing technology, and the capacity of the laser components on the destructive threshold. The thin-film components (substrate + thin-film) that we have produced have better mechanical strength. The transmittance of those components with two-sided AR coatings is greater than 97% (the residual reflection is about 2%, according to theoretical calculations), and the absorptance is less than 1%; the transmittance of those components with single-sided AR coatings is in the range of 63-64% (see Fig. 1). Under the effects of a TEA laser with a pulse width of 100 ns and an energy density greater than 2 J/cm^2, the thin-film components are still not damaged after nearly 10,000 strikes.

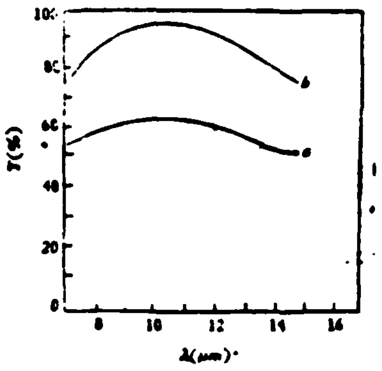


Fig. 1 The transmittance vs. wavelength curve for Ge substrate with AR coatings on one side (a) and on both sides (b).

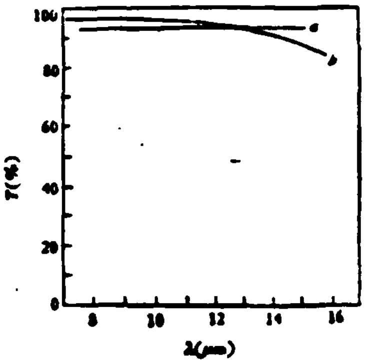


Fig. 2. The transmittance vs. wavelength curve for uncoated KCl substrate (a) and that with AR coatings on both sides (b).

We also trail produced an AR coating for KCl window and its transmittance is increased from 92% to 95% (see Fig. 2). Its moisture resistance capability has also been greatly improved and it is suitable for use in a normal environment.

II. Investigation of Increasing the Ability of Ge AR Coatings to Resist the Destructive Effects of Laser Radiation

The destructive threshold of Ge is low. Therefore, in the IR range, especially at the mid-IR, KCl and NaCl are commonly selected as substrates. But for the CO_2 laser, a Ge substrate coated with a ZnS AR coating, is still widely used as the output window. The structure of ZnS coating is cubical, pillar-shaped with voids in it; therefore, it peels off easily when exposed to moisture. Earlier, foreign research produced an excellent coating technology by which the films coated would not peel off even when boiled in 5% salt solution. Due to equipment limitations, we employed the technique of adding a Ge covered lining between the film and the substrate to increase its overall strength. Film made in this manner still maintains good adherence after it is placed in boiling water for 5 minutes. However, this film quickly breaks down under the effects of a high-current, pulsed high-power CO_2 laser. We conducted analyses and tests, and concluded that the damage was caused by the Ge lining's absorption of laser energy. Under the same laser effects, the threshold of ZnS film without the Ge lining is about 10 times higher than that of the one with the lining. It is obvious from this that the improvement of mechanical adherence of a thin-film does not imply that the destructive threshold will rise. The destructive threshold is mainly determined by the substrate raw material, the heat absorption effect caused by defects resulted from machining and thin-film manufacturing technique, and the character of the operating laser, etc. The damage to the AR coating usually occurs at the interface between the substrate and the film, and the main factor that will govern the destructive threshold is the substrate [2]. Now, our work -- with regard to the main factors that affect the destructive threshold -- is discussed as follows:

1. The Effects of the Substrate

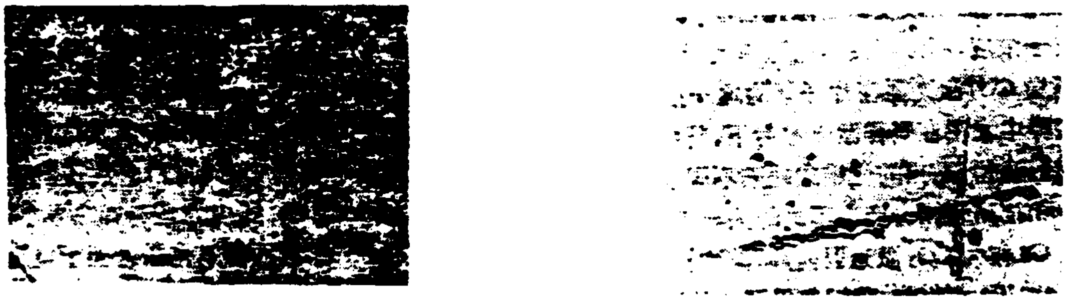
(1) The Effect of the Substrate Raw Material Properties

The lattice vibration absorption band of most IR semiconductor materials extends over a wide wavelength range. There is no problem in their use at 10μm and this holds for Ge. The reasons for limiting Ge to long wavelength use are crystal growth technology, impurity absorption of the raw material and free carrier absorption. This impurity absorption is generally distinctive only at low temperatures, and will not be considered here. The free carrier absorption is proportional to the square of the wavelength and also to the square of carrier concentration. The purity and chemical composition of the raw material affect, to a large extent, the carrier concentration and, therefore, affect the absorption. Further, the annealing speed during controlled crystal growth causes the various stress conditions and can change the material's ability to withstand laser radiation [1]. It is generally thought that the Ge chip should have the highest possible purity and the lowest possible carrier concentration; therefore, Ge chips with a resistivity greater than 25Ω-cm are commonly used. In practical work, we found that resistivity is not the only parameter to use in evaluating the window of CO_2 laser. Resistivity greater than 25Ω-cm is not necessarily the best resistivity. In the laboratory we found that under the same laser radiation, the ability of Ge chips, with the same resistivity, to withstand the pulsed laser strikes differed from sample to sample by a factor on the order of thousands. It is our opinion that differences in crystal lattice displacements or defects will cause different carrier concentration and thus, the absorption rate. Differences in stress and crystal structure will result in different thermal conductivities which are closely related to the ability of Ge to resist damage. Therefore, if we use Ge as the window of the CO_2 laser, its lattice displacements and the particular manufacture growth technology can not be overlooked. As to what the best resistivity is, it is currently inconclusive. Consequently, we chose Ge chips with 4 different resistivities to conduct tests on destructive thresholds and labeled them #2, #3, #4, and #5 (with resistivities 5-5.5Ω-cm; 9-10Ω-cm; 18-20Ω-cm; and 50Ω-cm, respectively). The raw material parameters and quality of polish of these 4 Ge chips are basically the same. Under the same laser pulse (energy density >1 J/cm^2, pulse length <1μs), the extent of damage is, in ascending order, #3, #4, and #2 and #5. This coincides with the results of reference [3], because sample #3 had the least absorption at 10μm. The optical micrographs of these 4 Ge chips are shown in Fig. 3.

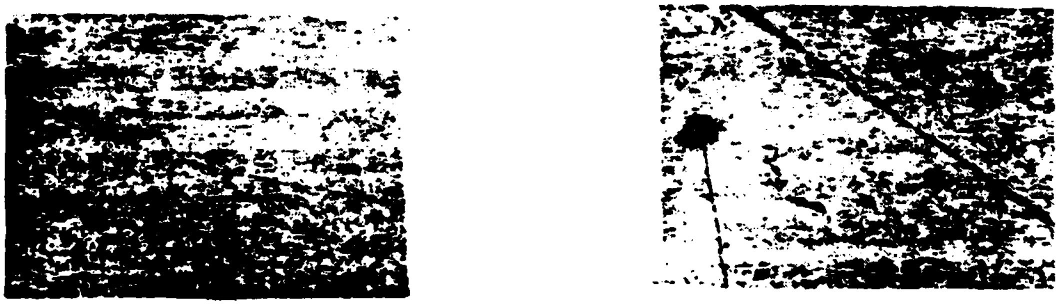
It can be seen from the pictures that Ge chips with different resistivities result in, under the same laser radiation, different degrees of destruction.

before radiation (a) #2 after radiation

before radiation (b) #3 after radiation



before radiation (c) #4 after radiation



before radiation (d) #5 after radiation

Fig. 3. Optical micrographs before and after laser radiation (25x). The black spot in picture (d) is the point of laser radiation.

(2) The Influence of Surface Roughness and Polishing Defects

The metamorphic layers are formed during machining of substrate, and thus create stress. The inlayed polisher will increase absorption. The radiative electric field of laser is markedly enhanced by the defects of machining, especially at deep and narrow scratches the radiative electric field increases as a function proportional to the square of substrate's index of refraction. Such regional increases of the electric field decrease the destructive threshold as a function proportional to the fourth power of the substrate's index of refraction [1]. In general, the roughness and the penetration intensity of electric field have the following relation: $E_{TH}\sigma^{0.61}$= constant, where σ is the root-mean-squared roughness and E_{TH} is the penetration intensity of electric field. From this we can see that the rougher the surface, the weaker the penetration electric field, i.e. the lower the destructive threshold. Furthermore, it can be seen from the after radiation picture of sample #5 that, although the laser did not directly hit the scratch, the damage nonetheless developed along the scratch. Dust on the substrate will enlarge cracks in the thin-film and loosen its structure; and therefore will affect its absorption characteristics causing the destructive threshold value to drop.

2. The Effects of Coating Quality

The effects of the quality of AR coating on the destructive threshold are more complicated. Strictly speaking, it is necessary to peel the thin-film and isolate each factor of influence, then investigate them one by one. Here, we only propose a few rough opinions.

The AR coating material should have both good optical and mechanical properties. It should also be compatible with the substrate. Based on different requirements, single-, double-, and triple-layer film systems can be each selected to obtain the best antireflective efficiency.

Second, the technology of coating application is also an important factor that would affect the threshold value. We tested the following several factors:

(1) The Effect of Steam's Angle of the Incidence during Steam Coating

The molecules of the coating material have greater kinetic energy in the perpendicular direction. Therefore, the thin-film formed by perpendicular coating deposition is firm and dense, and thus resulting in a high destructive threshold. However, thin-film made at large angle of incidence will have large stress, and thus low threshold value.

(2) Baking Temperature of the Substrate

150°C is a good baking temperature for Ge chips with ZnS coating. At this temperature, the rate of evaporation is high; the coated film is dense and well-adhered with high threshold value. If the baking temperature is too low, the coated film's index of refraction is rather high, and residual reflection will increase. If the baking temperature is too high, the coated film will be misty which affects its transmittance and results in high absorptance as well as low threshold value.

(3) The Rate of Deposition

If the rate of deposition is low, the crystal grains of the thin-film large, and absorption and light scattering are both large. If the rate is high, the density at the curing center is high; the grains are fine, and the film is well adhered. But, if the rate is too high, the molecular transport rate of the thin-film will drop, thereby causes the stress to increase and the wavelength stability of the film to decrease. The appropriate rate is 500-600 Å/min.

(4) Residual Gas

The residual gas in the vacuum chamber, especially the residual moisture greatly affects absorption, and therefore the threshold value. Automatic monitoring has been realized abroad. At Hughes Laboratory in the United States, when coating ThF_4 film, since the residual moisture has been reduced to a minimum, the absorption coefficient has been lowered from 10-20 cm^{-1} to 1 cm^{-1}. The effects of moisture on ZnS film can be even greater.

(5) The Distribution of Standing Wave Field in the Thin-Film

The maximum and minimum values of the standing wave field and its distribution in the thin-film directly affect the threshold value. For example, in the destructive threshold experiments of Ge with AR coating performed cooperatively by B. E. Newnam and D. H. Gill, when comparing the destructive threshold values of the substrates, with and without coating, it was discovered that the ratio of two destructive threshold values was very close to the ratio of the square of electric fields under those two conditions. This illustrates that the damage threshold and the intensity of field are closely related. This explained the close relationship between the destructive threshold and the field intensity. This is an important reference for conducting film system design.

3. The Effects of the Character of the Laser

Currently, there are several theoretical models of the penetration process and they all involve the characteristics of the laser apparatus - e.g. wavelength, pulse sustaining time, and diameter of laser beam mark, etc. Generally speaking, when the thin-film component is under the radiation of laser pulses with width shorter than 100 ns, electronic avalanche is the main cause of damage [1]. Since the high electric field associated with short pulses can create $10^{18}/cm^3$ free electrons, this is the condition for avalanche to occur. Therefore, it is necessary to strictly control the defects in substrate and film when operating under short pulses. For long pulse radiation, the destruction of the thin-film component is directly proportional to absorption.

Furthermore, for lasers of equal power density, the ones with larger beam diameter are more likely to cause damage than those with smaller beam diameter because the probability for the radiation of a laser beam of large diameter to catch defects is greater.

III. IR-Range Antireflection Film of KCl, NaCl Windows

With high IR transparency, low reflection loss and high destructive threshold, KCl and NaCl are very good materials for making IR windows. They are especially suitable for making the window of a high-current, pulsed IR laser. Their transmittance is 92% at wavelength in the range of 9-16μm. But KCl and NaCl deliquesce easily. A method most commonly used abroad for preventing deliquescence is to put a low electric current around the window. We, however, employed the film-coating method. CaF_2 film was coated on both sides of a KCl chip (the index of refraction of CaF is 1.3). The residual reflectance after the coating was applied was about ~~2-3%~~ 1% and transmittance was raised by 2-3%. A KCl lining of 2/5 the maximum thickness was also coated between the film and the substrate to increase strength. The substrate was sealed on all sides by a moistureproof glue to isolate the entire KCl surface from the air. After such treatment and having been exposed under 90% humidity and temperature greater than 30°C for 10 days, the coated component remained undamaged.

KCl can also be coated with an As_2S_3 protective film. There are people abroad who applied $Ge_{45}Se_{55}/As_2S_3$ as double-layer AR coating and achieved a destructive threshold up to 1-2 J/cm^2. The $As_2S_3/ThF_4/As_2S_3$ triple-layer AR coating reported in reference [1] took not only the optical properties into account but also the distribution of the standing wave field; thus, it not only increased the destructive threshold but also achieved a moistureproofing effect.

There have been many studies abroad on the destruction mechanism of KCl and NaCl. Reference [4] considered the mechanism as follows: under a low intensity (250-800 MW/cm^2) laser radiation, the change in their absorptance is reversible. The absorptance remains unchanged under an intensity ranging from 800 MW/cm^2 to 3-5 GW/cm^2. When the intensity exceeds 3-5 GW/cm^2, the change in absorptance becomes irreversible and permanent damages appear in the material. Therefore, the lowest intensity I_F under which change in absorptance becomes irreversible is defined as the destructive threshold of the material. Under the radiation of this intensity, microscopic damages begin to appear gradually. As the intensity of the laser increases, these microscopic damages interact with the laser, thus absorbing more energy from the light field, and the microscopic damages gradually accumulate and expand. When the intensity reaches 7-10 GW/cm^2, penetration occurs. There is a visible spark accompanying the occurrence of penetration, which blocks the laser radiation and creates microscopically detectable residual destruction.

Generally speaking, the theoretical linear absorptance of KCl and NaCl is 10^{-5} cm^{-1}. Due to the existence of impurities and defects, the actual linear absorptance is 10^{-3} cm^{-1}. Their destructive thresholds are $I_{FNaCl}=3.5$ GW/cm^2 and $I_{FKCl}=6$ GW/cm^2, respectively.

During the course of experiments, Room 401 of Institute of Colored Metal Study, and Room 14 of Institute of Mechanics, Academia Sinica have given us great help. We would like to express our appreciation to both offices here.

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