PHOTOCHROMIC AND PHOTOREFRACTIVE

EFFECTS IN BISMUTH SILICON OXIDE

DOPED WITH IRON AND BISMUTH

SILICON OXIDE DOPED WITH

IRON AND GALLIUM

By

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Bachelor of Science
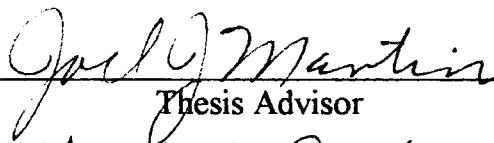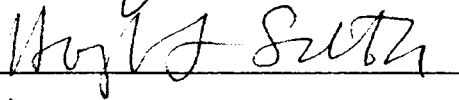
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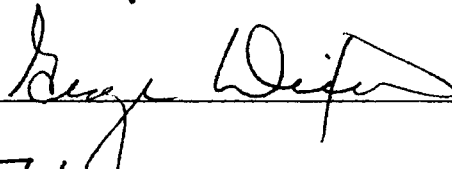the Degree of
MASTER OF SCIENCE
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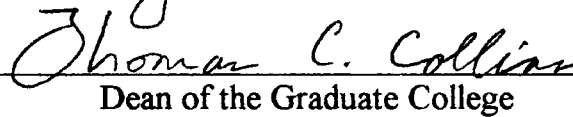SILICON OXIDE DOPED WITH

IRON AND GALLIUM

Thesis Approved:

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Thesis Advisor

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Dean of the Graduate College

ACKNOWLEDGMENTS

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Thanks goes to Mr. Charles Hunt for his infinite patience in teaching me how to grow good samples. I also wish to extend to Dr. Larry Scott a special thank-you for taking time from his busy schedule to serve on my graduate committee.

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CHAPTER 1

INTRODUCTION

Bismuth silicon oxide, $Bi_{12}SiO_{20}$, and Bismuth germanium oxide, $Bi_{12}GeO_{20}$, hereafter referred to as BSO and BGO, are members of the sillenite family of crystalline compounds. These unique crystals have considerable potential for a wide variety of technological applications, namely, optical data processing[1-3], real time interferometry[4], nonlinear optical applications[2,5], and as storage media in holographic memory systems[6,10]. These materials exhibit a large photorefractive effect, a change in the refractive index induced by light[6-9], which supports a large number of emerging applications. For this reason, BSO and BGO, are being studied extensively. A necessary preliminary step in the understanding of these materials is to characterize the defect structures of these materials.

BSO and BGO are closely related materials. Both have the same body-centered cubic structure, first studied by a Swedish chemist named Sillen[9], and have a symmetry that correspond to the m3 point groups, which lack inversion symmetry[9,11]. Room temperature studies of BGO were undertaken by Abrahams et al.[12]. The unit cell consists of a Ge/Si atom surrounded by four oxygens in a tetrahedral geometry. This combination occurs at all eight corners of the cube and at the center of the cube. Bismuth atoms within the cube have seven oxygen atoms arranged in a distorted octahedral geometry around each[9]. Due to the lack of inversion symmetry, BSO is piezoelectric and electro-optic. These electro-

optic properties, in addition to the defects inherent in the crystal, combine to produce the photorefractive effect.

Defects

A serious problem in the production of these crystals is that the internal defect structure, a combination of electrons or holes trapped at impurities and other defects[13], differs depending on the source of the crystals. Quality crystals have been produced by growth in air using the Czochralski method, producing crystals with a yellow coloration[14], and hydrothermal techniques; resulting crystals are colorless and have a much lower trap density than Cz materials[15]. BSO crystals are normally grown using the Czochralski method. The resulting yellow color is attributed to the presence of a deep electron donor, possibly a bismuth occupying a silicon site[14]. This anti-site bismuth would act as a donor. Hou et al[16] postulate that the donor is due to an electron occupying a silicon vacancy complex. Oberschmidt[17] originally argued on the basis of enthalpy measurements for the improper occupation rather than a vacancy. More recent work by Craig and Stephenson[18] support this possibility. They found that the compound $Bi_{25}FeO_{40}$ has the same structure as BSO with Fe on one Si site and Bi on the other in the unit cell.

Photorefractive Effect

The electrons (or holes) trapped at the impurities can be excited into the conduction (or

valence) band by exposing the crystal to light. Once in the conduction band, the electrons migrate until they recombine at an empty shallower trap. Figure 1 shows an energy level diagram depicting the proposed charge migrations that occur when BSO is illuminated with near band edge light. Exposure at low temperatures to band edge light produces photochromic absorption bands. BSO doped with iron or BSO doped with iron and gallium each contains unique acceptor states that create the absorption shoulders found in their photochromic curves. Figure 1, (b) and (c), also shows proposed energy levels for these acceptor states. If the exposure to light is nonuniform, such as in a four-wave mixing experiment using crossed laser beams, a nonuniform charge redistribution results. This distribution of charge produces a complementary electric field pattern within the crystal. In noncentrosymmetric crystals, like BSO, the field produces a corresponding pattern in the index of refraction through the electro-optic effect[6,8,14]. This pattern in the index of refraction, or grating, is known as the photorefractive effect. The lifetime of the grating depends largely upon the material, but also on the temperature, and the wavelength and intensity of the "write" or exposure beam.

In addition to undoped BSO, recent studies have included BSO doped with 2% Fe and also BSO doped with 2% Fe and 5% Ga. The purpose of the dopants is to provide information on the compensation of the deep donor through the resulting changes in the absorption spectra and the changes in the charge distribution in the photrefractive effect.

C.B. ‾‾‾-e‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾‾

(a)　　　3.1eV　(b)　　　　　(c)

3eV　　　　　　　2.9eV

3.2eV　　3.45eV

Donor　　　Fe　　　　Ga

V.B. ‾‾‾
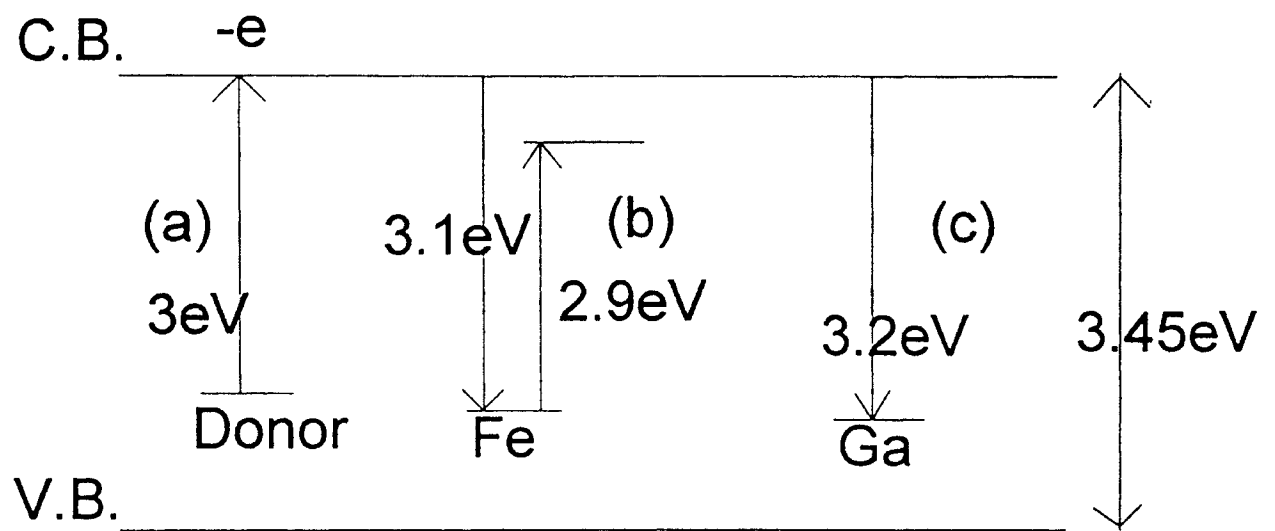
Figure 1.   Energy level diagram showing the proposed transitions responsible for the

photochromic bands and the photorefractive data.

Statement of Purpose

BSO:2% Fe and BSO:2% Fe 5%Ga form photochromic absorption bands when exposed to 3eV, near band edge, light. In four-wave mixing experiments, both crystals exhibit the ability to form gratings. In the following experiment, a systematic study of the low temperature absorption bands, the isochronal anneals, and the physical properties of the photorefractive gratings for both BSO: Fe and BSO: FeGa are presented. Comparison of the absorption bands and the photorefractive data help to characterize the defect structures of these crystals. Ultimately this information will bring a better understanding of BSO and allow for utilization in technological applications.

CHAPTER 2

EXPERIMENTAL PROCEDURE

For this study two doped samples were grown at Oklahoma State University

using the Czochralski method. The first was a 2% iron-doped BSO sample. Iron

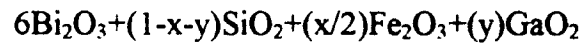doping was accomplished by adding iron-oxide to the melt with a corresponding reduction

in SiO_2 by the following ratio:

$$6Bi_2O_3 + (1-x)SiO_2 + (x/2)Fe_2O_3$$

The second sample to be grown was doped with 2% Fe and 5% Ga by the following

formula:

$$6Bi_2O_3 + (1-x-y)SiO_2 + (x/2)Fe_2O_3 + (y)GaO_2$$

The chemicals used were Johnson-Matthey Grade-1 bismuth oxide (Bi_2O_3) and silicon

oxide (SiO_2).

Sample Growth

Both chemical mixtures were prepared for the melt by undergoing first, a mixing

period in a glass container for 48 hours, then the mixture was transferred to a

platinum crucible and heat treated at $800°$ C surrounded by a flowing oxygen

atmosphere for another 48 hours. Upon removal, the platinum crucible containing

the mixture was placed in a 20 kW 450 kHz RF induction furnace where it was heated to

approximately $900°$ C. Once the mixture was melted, an orientated seed crystal of

undoped BSO was dipped into the melt and slowly withdrawn. The seed was rotated at

30 to 86 revolutions per minute to prevent the formation of a darker center or core[19]. The

temperature of the melt was monitored by an Acufiber model 10 controller that uses a sapphire sensor in contact with the base of the crucible. The temperature set points, the temp-time controlled growth period, and the warm-up/cool-down ramp were controlled by an HP-86 microcomputer. The seeds for each of the two samples were [100] oriented Oklahoma State University Czochralski grown BSO. At the end of the growth period, the newly grown crystals were cooled to room temperature over a period of 48 hours to prevent cracking due to thermal stress.

Optical samples 1-1.5 mm thick were cut from the crystals. Both (100) and (110) surfaces were cut. Surfaces 90° degrees to the c-axis were used for absorption measurements, samples cut 45° degrees to the c-axis were utilized for photorefractive measurements.

Absorption Measurements

For the absorption measurements, the samples were mounted on the cold finger of a CTI closed-cycle cryogenic refrigerator at a 45° angle to the sample beam of a model 330 Perkin Elmer spectrophotometer. The 45° angle allowed the polished samples to be exposed to the excitation light beam without having to remove the cold-head from the spectrophotometer. All optical absorption scans were made at 9-15 K. Before scanning, the samples were exposed to near band edge light, at 8-15 K, using an Oriel 200-W Xe lamp and a Spex Minimate monochrometer with 5 mm slots. The near band edge light "writes" photochromic bands in the samples. The scans using the spectrophotometer are used to optically characterize the samples in their as-grown conditions.

Then, the thermal stability of these photochromic bands are tested by performing a series of isochronal anneals. After collecting an absorption spectrum at 8-15 K the temperature of the sample, using the refrigerator, is raised to the desired value and then returned to 8-15 K for the next absorption scan.

Photorefractive Measurements

The samples cut 45° degrees to the c-axis were used in an experiment designed to measure photorefractive effects. A 442 nm Helium Cadmium laser beam was split in a beam splitter. The resulting two beams were bent back towards the cold finger mounted sample. When the beams reached the sample they were crossed to form a grating inside the sample. The sample was oriented perpendicular to the incoming beams. The resulting grating was "read" by a 632.8 nm Helium Neon laser. This "read" beam was geometrically oriented at the Bragg angle, approximately 9°, and the resulting refracted beam was fed into a photomultiplier tube, channeled into a Hewlett Packard digital scope and a Keithley Digital Multimeter, and finally analyzed by an IBM 286 personal computer. The Helium Cadmium beam, or "write" beam, is sent through a shutter before it hits the beam splitter. This allows the "write" time to be predetermined. Other predetermined variables are: the intensity of the "write" beam, the intensity of the "read" beam, through use of various neutral density filters, and the temperature of the sample by using a CTI Cryogenic refrigerator. A schematic diagram of the experimental setup is found in Figure 2.
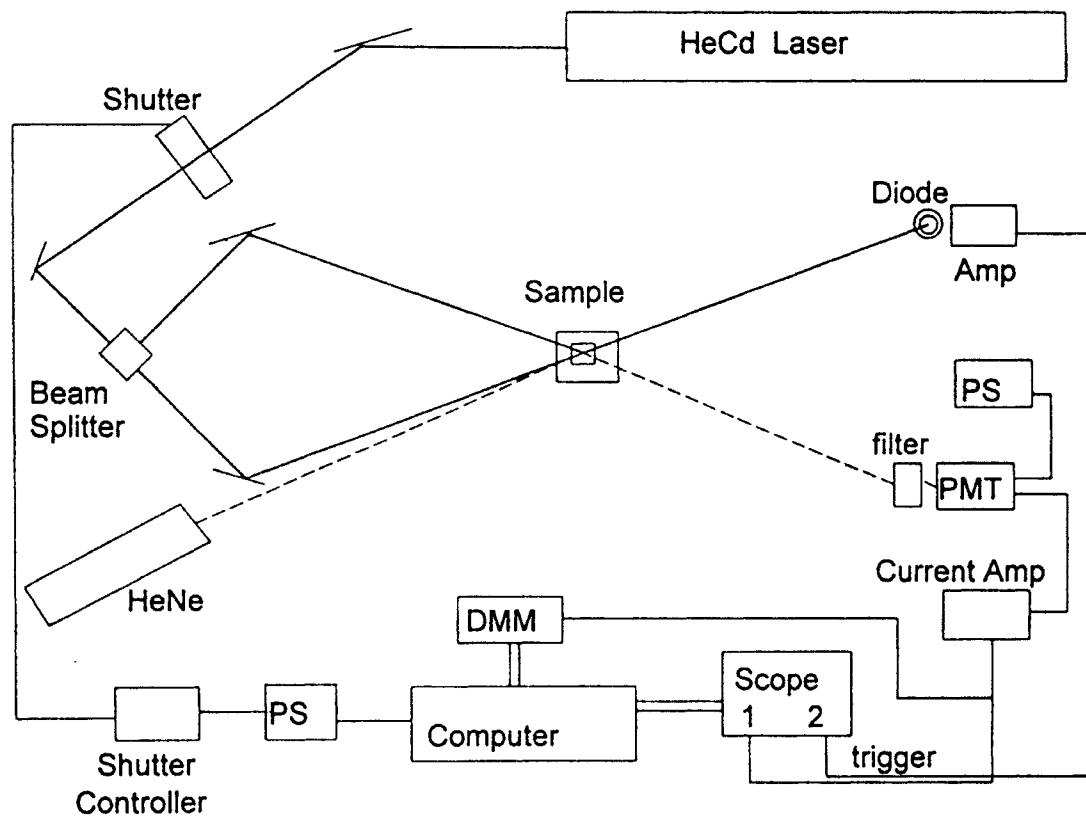
Figure 2. The experimental setup for measurement of photorefractive effects in

BSO:2%Fe and BSO:2%Fe5%Ga.

CHAPTER 3

RESULTS AND DISCUSSION

Photochromic Curves

Figure 3 shows absorption spectra taken at 10 K on undoped, Al-doped, Fe-doped, and FeGa-doped samples. These curves serve as the baselines or background for the photochromic data. The important differences between these samples occur at the absorption shoulders around 3.3 eV for Al and near 3 eV for the other three curves. The Al-doped sample is a nearly colorless crystal. The dopant occupies the Si site and acts as an acceptor to compensate the deep donor (see figure 1, (a)) and thus removes the yellow coloration[20]. The curve for the undoped sample shows the absorption shoulder near 2.9-3 eV which causes the yellow color typical of BSO and BGO. The shoulder is thought to be due to excitations from the deep donor, in Fig. 1, to the conduction band. This shoulder is missing (or at least greatly reduced) in the Fe, Al and FeGa-doped specimens. The absorption cut-off in the Al-doped sample is at 3.3 eV. Hart *et al.* [20] found the Ga-doped BGO also cut-off at 3.3 eV. These results suggest that the Al and Ga acceptor levels are about 3.3 eV below the band edge. Doping with Fe and with FeGa, also removes the yellow color, but the absorption cut off is between 3 eV and 3.2 eV. Since the FeGa-doped sample had enough Ga to fully compensate the donor, its cut-off would be expected to be near 3.3 eV[20]. Thus, it appears that the cut-off is due to an internal transition in the Fe, perhaps from the spin 5/2 ground state to the spin 3/2 excited state.
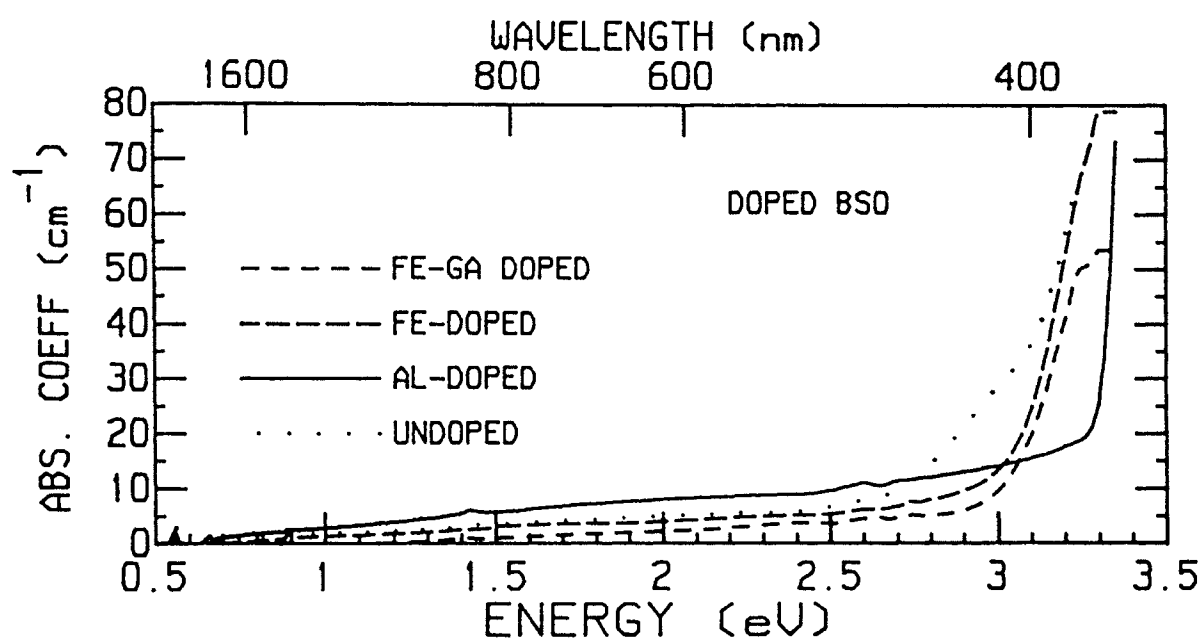
Figure 3. Comparison of the baseline photochromic bands of undoped, Al-doped,

Fe-doped, and FeGa-doped BSO.

This excited state would be located within the band gap, but would be very close to the conduction band[21]. This could possibly allow for thermal excitation at room temperature of these electrons into the conduction band. Thermal excitation may account for a winking phenomenon noticed in both the Fe-doped BSO and FeGa-doped BSO samples. This will be discussed in more detail later.

Figures 4 and 5 show absorption curves in Fe-doped and FeGa-doped BSO after irradiation using 3eV light from the Oriel 200W Xe lamp. The irradiation was applied in increments up to a total of 60 minutes for the Fe-doped sample, and up to a total of 90 minutes for the FeGa-doped sample. There was a noticeable growth in signal strength in the shoulder region in both samples.

In figures 6 and 7, the baseline was subtracted from the absorption curves in figures 4 and 5 and the resulting change in absorption for each illumination time was graphed. The growth in absorption for the Fe-doped sample is not centered at a specific energy. Instead, there is a spread of energies from 2.5 eV-3.2 eV that are affected. There is also a small peak at 1.4 eV. This corresponds to the results of Hart *et al.*[20]. BGO:4%Ga has a peak at 1.55 eV and BGO:5%Al has a peak at 1.38 eV. These two also have a broad, relatively featureless band centered around 2.45eV. The Fe-doped band appears to be centered closer to 2.8 eV.

In addition to these peaks, BGO: Al and BGO: Ga each have a characteristic peak at 1 eV, and 1.1 eV, respectively. These peaks are thought to be due to the addition of the dopants into the material. By analogy with quartz, the proposed mechanism is the hole center. The corresponding hole center peak in the FeGa-doped sample is absent. The

FeGa-doped sample however, has peaks at 2.2 eV and 3.3 eV but no peak at 1.4 eV. There is growth in the 2.5eV-3.2eV range that corresponds with the Fe-doped sample.

In figures 8 and 9 the isochronal anneal graphs are shown. These contour plots show the decay of the photochromic bands. In Fe-doped, the bands decay between 125K and 150K. In 4%Ga-doped BGO, the infrared bands (1.1 and 1.55 eV), and a major portion of the visible range anneals between 100 and 120 K. Al-doped BGO is similar except the anneal occurs in the 80-100 K range. The remaining visible range absorption decays just below 230 K[20]. In FeGa-doped, there seems to be only a small decay at 300K. No higher temperature studies were performed; however, future work should include higher temperature anneal studies on the FeGa-doped samples.
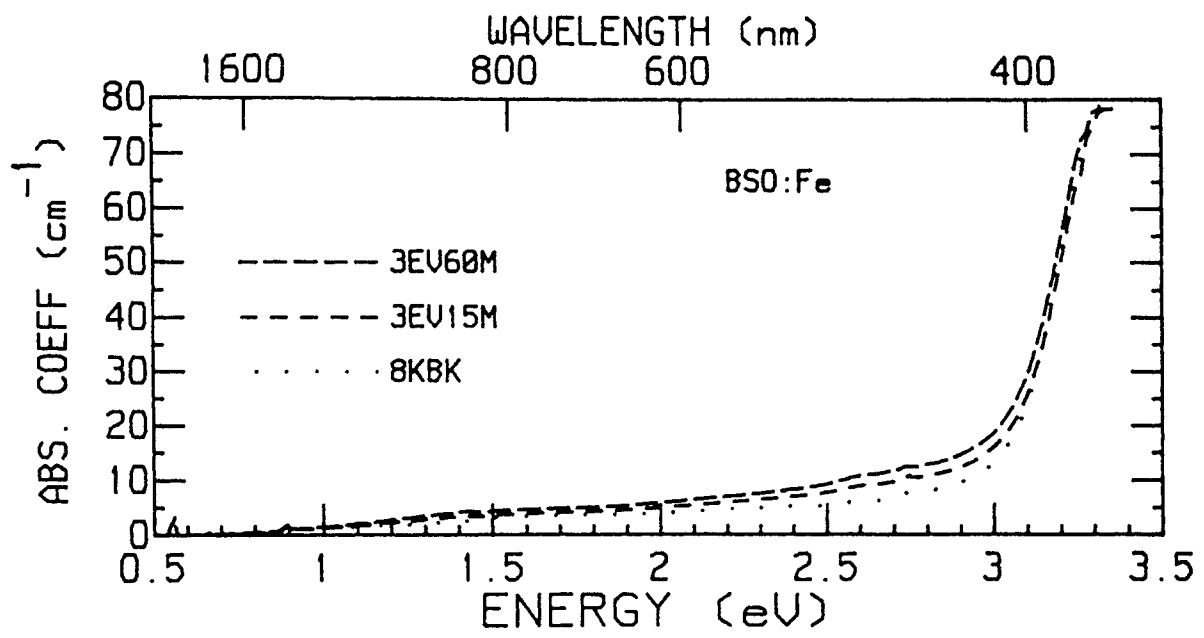
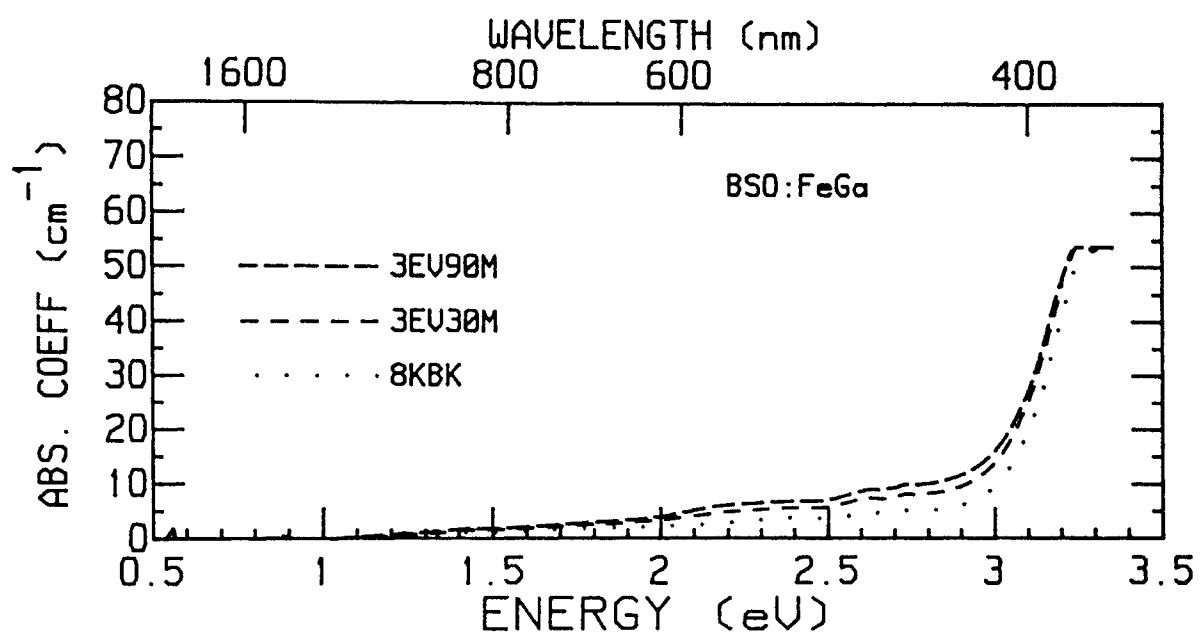Figure 4. Photochromic absorption bands for BSO:2%Fe

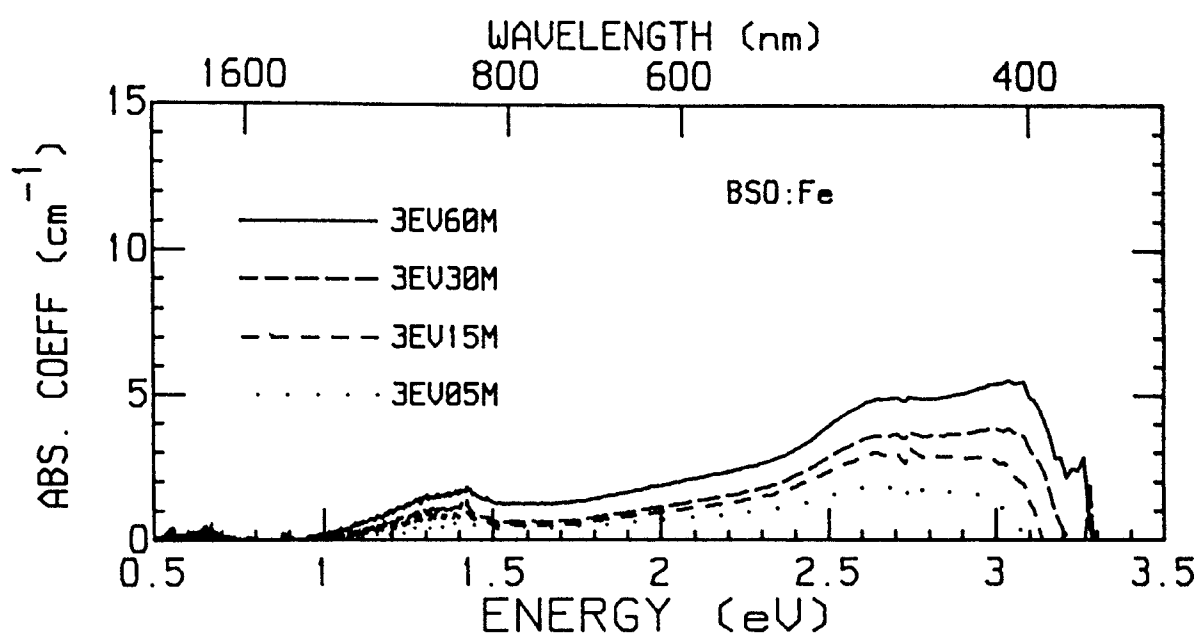Figure 5. Photochromic absorption bands for BSO:2%Fe5%Ga

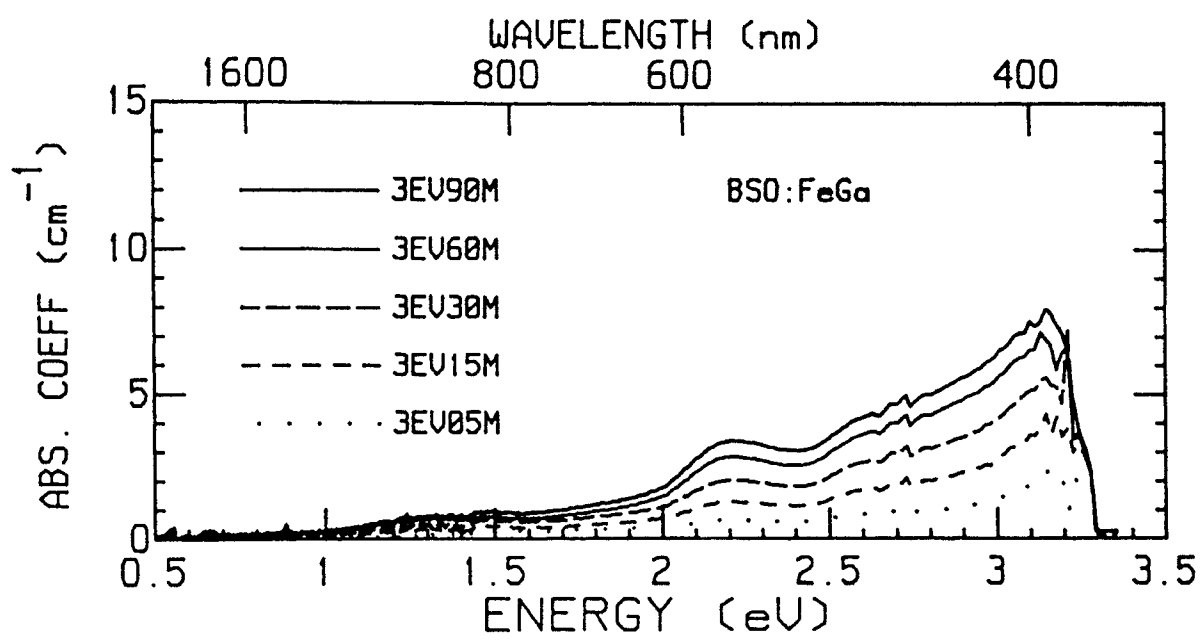Figure 6. The change in the absorption spectra for BSO:2%Fe

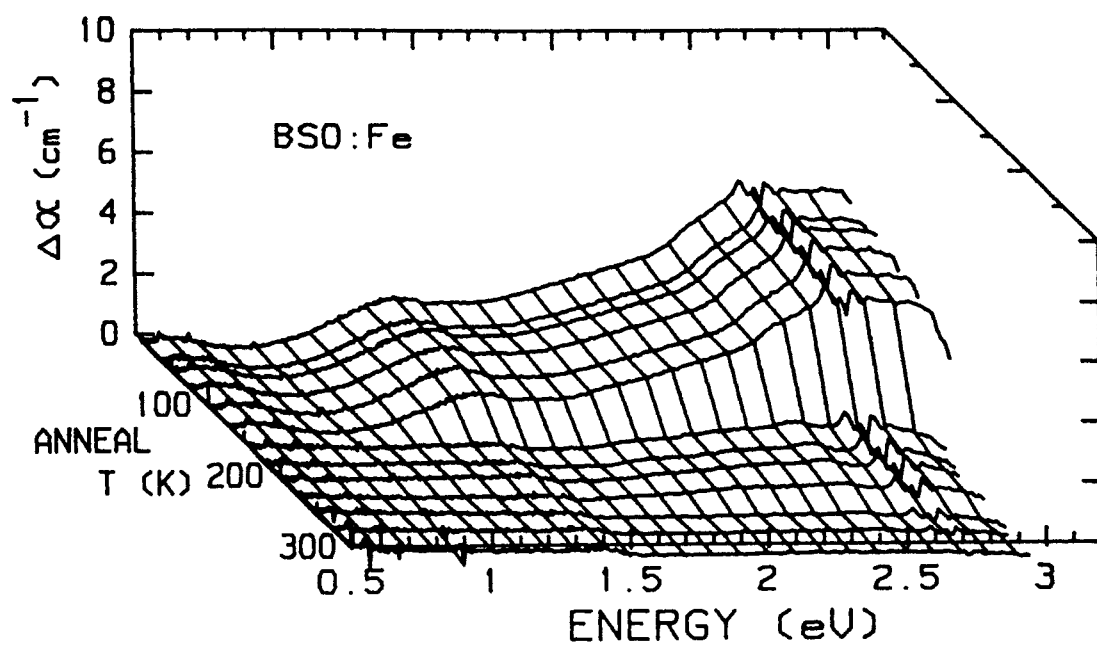Figure 7. The change in the absorption spectra for BSO:2%Fe5%Ga

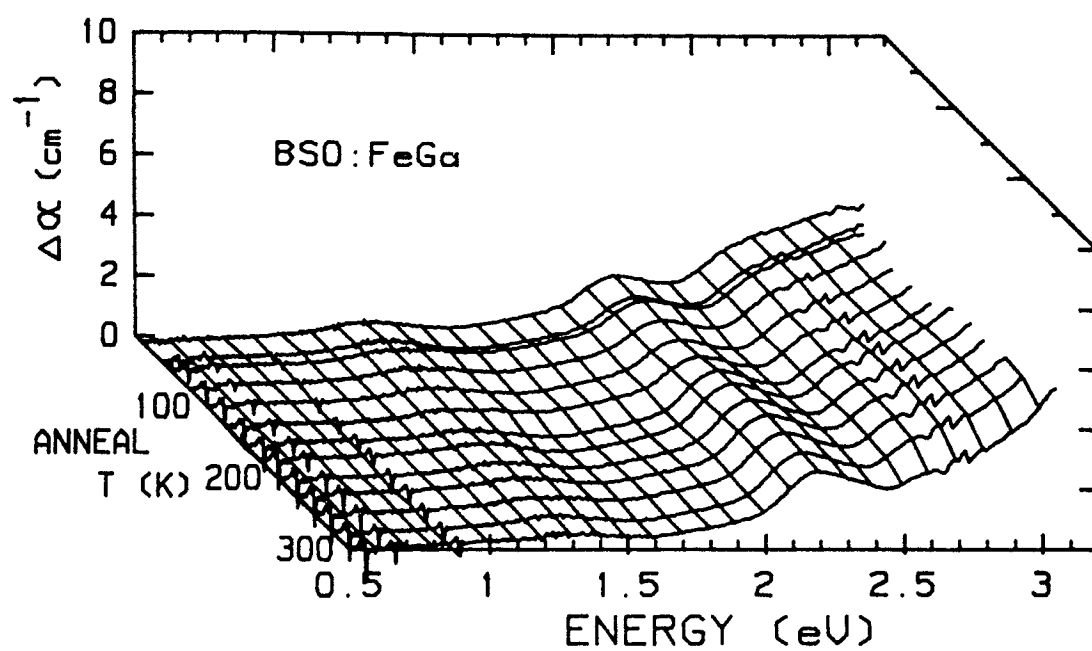Figure 8. The isochronal anneal contour plot for BSO:2%Fe

Figure 9. The isochronal anneal contour plot for BSO:2%Fe5%Ga

Photorefractive Data

Figure 10 shows the temperature dependence of the grating during the "write" process of BSO:Fe. The shutter time was 100ms. The grating spacing was measured at 2μ. At 300K there is an initial peak which decays through 200K. This leading peak may occur due to the increase in mobility at room temperature or thermal excitations. At 150K, however, there is a sudden rise in the observed signal. This rise correlates with the decay of the photochromic bands between 125K and 150K in Fig. 8. There is also a correspondence with the disappearance of the ESR Fe^{3+} signal[13]. Foldvari *et al.*[22] have found that the BGO:Fe photorefractive signal at room temperature has a different origin from the signal seen at low temperatures.

Figures 11, 12, and 13 show the temperature dependence of BSO:FeGa at different write times. Figure 11 was done with a shutter time of 500ms. Figures 12 and 13 are for the same shutter time of 2000ms. Figure 13 shows the first 500 ms of the 2000 ms data as a comparison for figure 11. The data between the two separate trials appear to be in good agreement. Note that the overall signal strength of the FeGa-doped sample is less than the Fe-doped signal in the first 100ms of the temperature dependence. The FeGa-doped response time, in terms of signal rise, was slower than that of the Fe-doped. Since both Fe and Ga are acceptors with nearly the same energy in the band gap, it is possible that they are "competing" for electrons. Also, the energy of our laser, 442nm, is not enough to create photochromic bands in Ga-doped BSO^{20}. It is possible that mainly the Fe is being affected during the temperature dependence "write" trials.

From Fig. 12, the sudden rise in signal can again be seen at low temperature. However, this time it appears at 100K. There is no direct correlation between this rise and the isochronal anneal decay of Fig. 9. There is also an initial peak at 300K which smoothes out by 225K. More study is needed to sort out the dynamics of the charge transfers taking place.

In addition to the temperature dependence studies, the winking phenomena mentioned earlier was investigated. The DVM was programmed to receive data for extended periods of time. This data was plotted on a personal computer real-time. The program used is reproduced in Appendix A. The plotted results are shown in Fig. 14. The winking may be attributed to the presence of iron in both of the samples[23]. The BSO:Fe sample showed the phenomena to a lesser degree than the BSO:FeGa sample. If indeed an internal ground to first excited state transition is occurring, the electrons in the excited state may be thermally excited into the conduction band at room temperature.

In Figs. 15 and 16, the change in the signal at room temperature for increasing shutter times is shown for both Fe-doped and FeGa-doped samples respectively. Both of the samples show the same trend to smoothly peak and then decrease in signal intensity. Fe-doped peaks at room temperature between 300-500 ms. FeGa-doped, however, shows max intensity at 500ms.

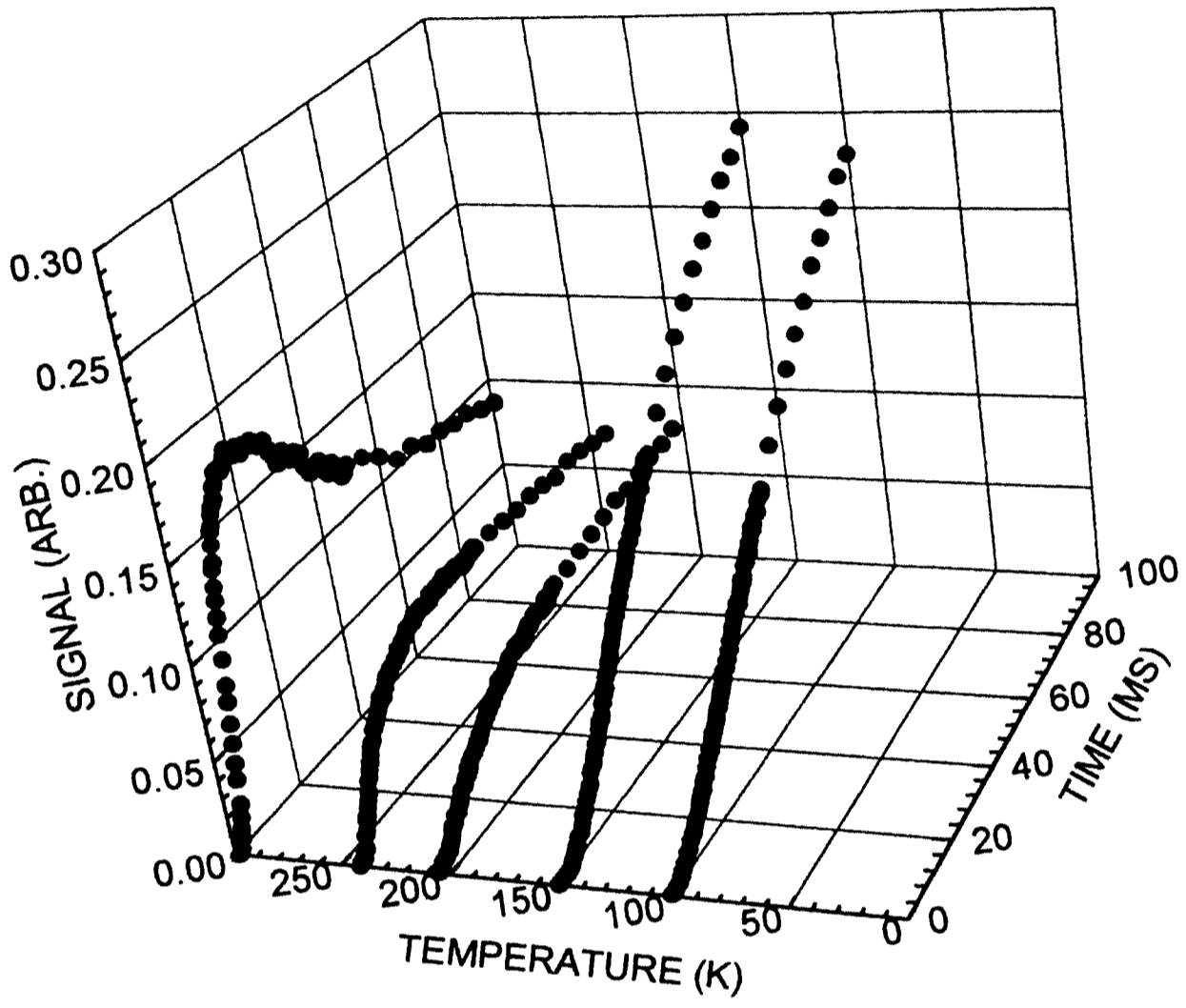Figure 10. The photorefractive temperature dependence of BSO:2%Fe

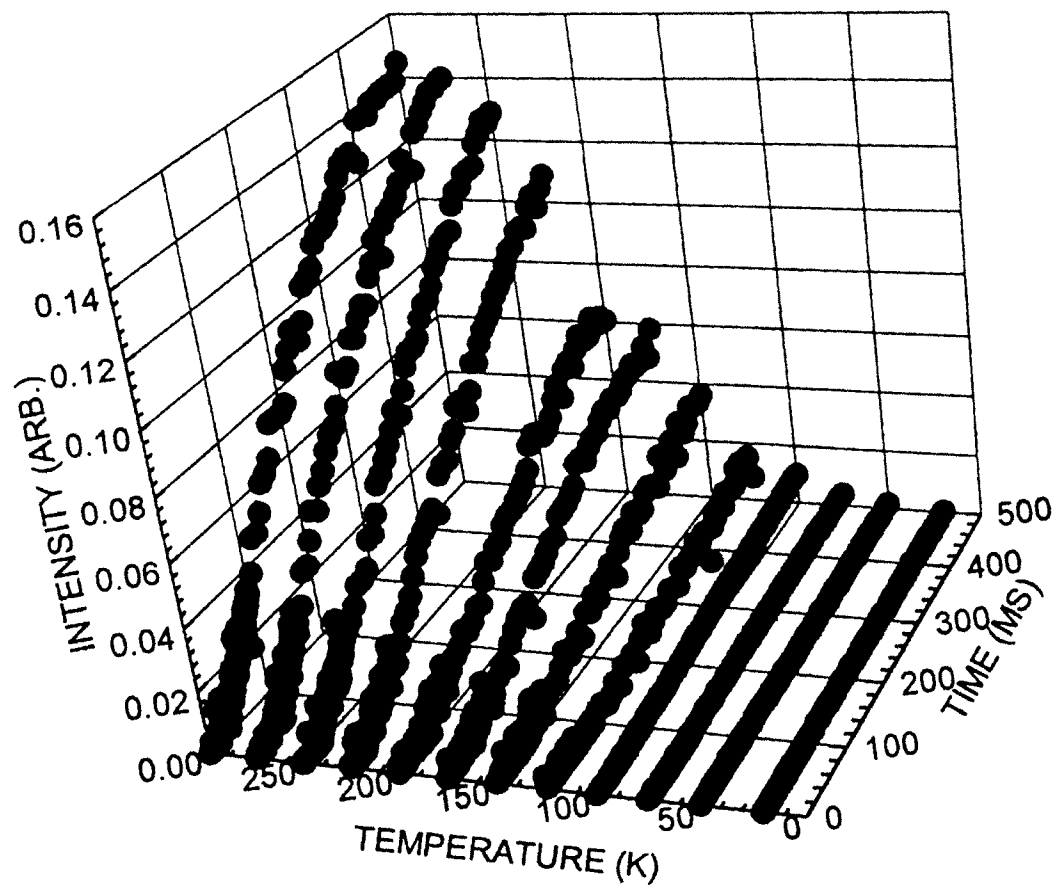Figure 11. The 500ms photorefractive temperature dependence of BSO:2%Fe5%Ga
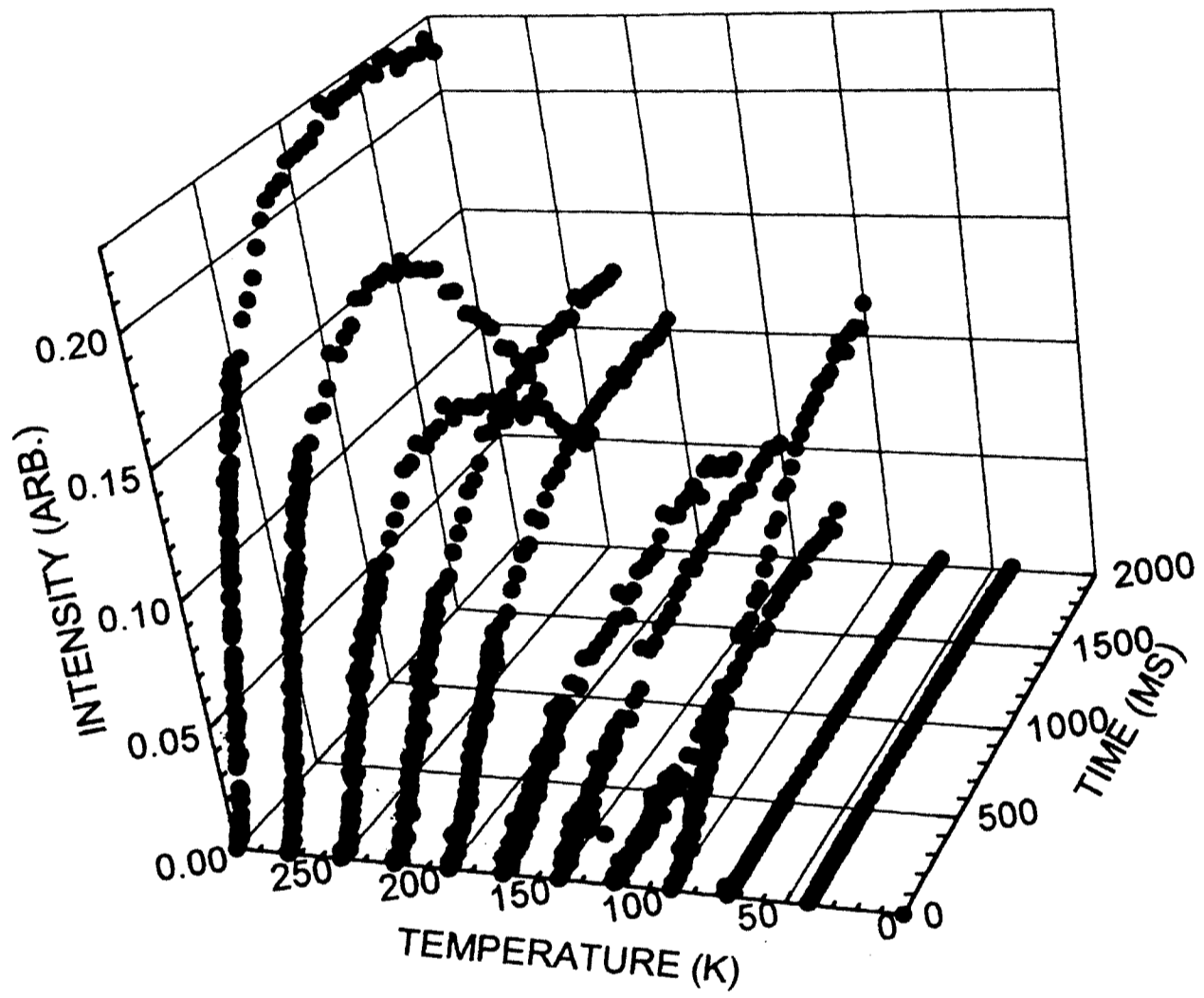
Figure 12. The 2000ms photorefractive temperature dependence of
BSO:2%Fe5%Ga.
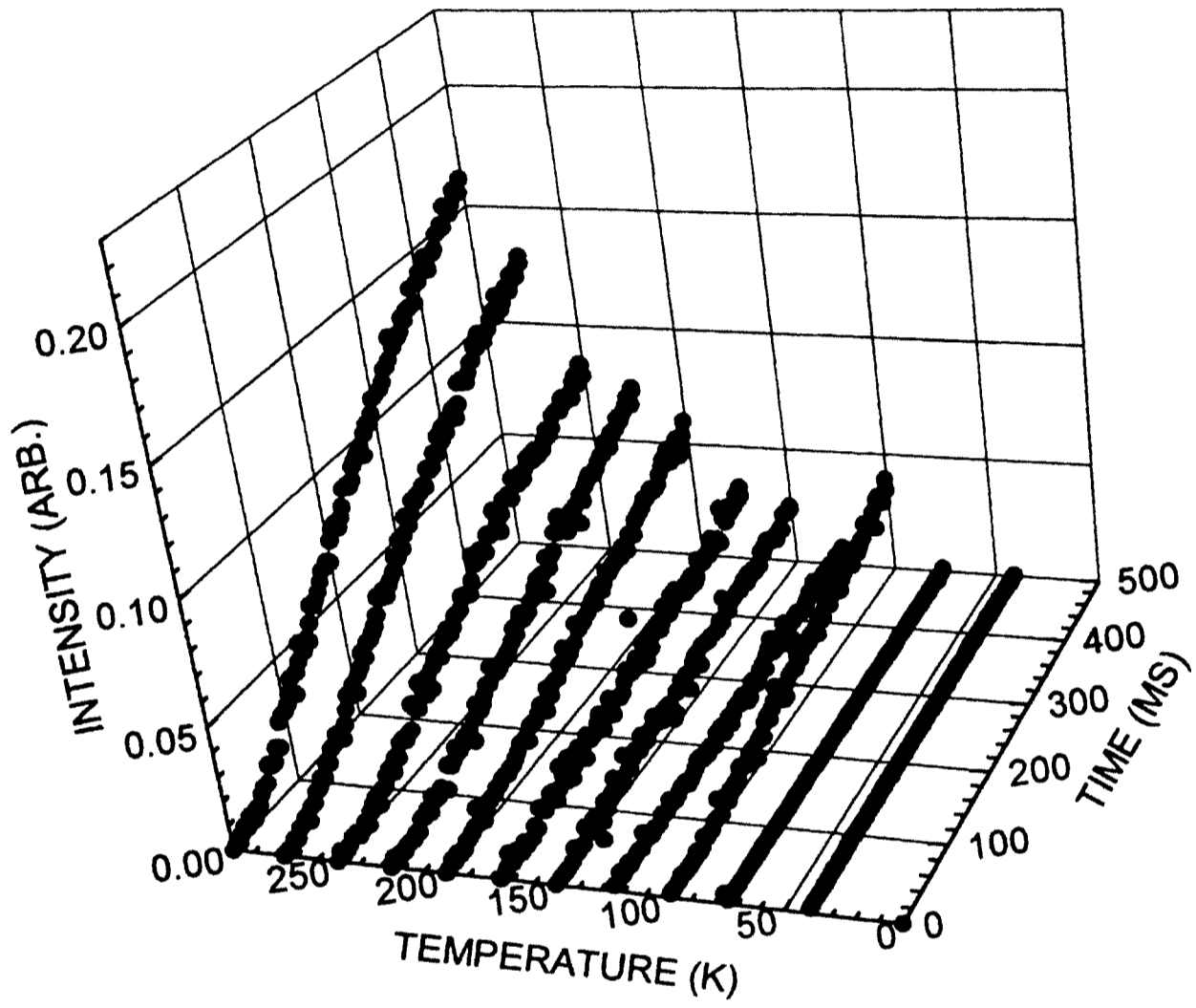
Figure 13. 2000ms temperature dependence rescaled to 500ms
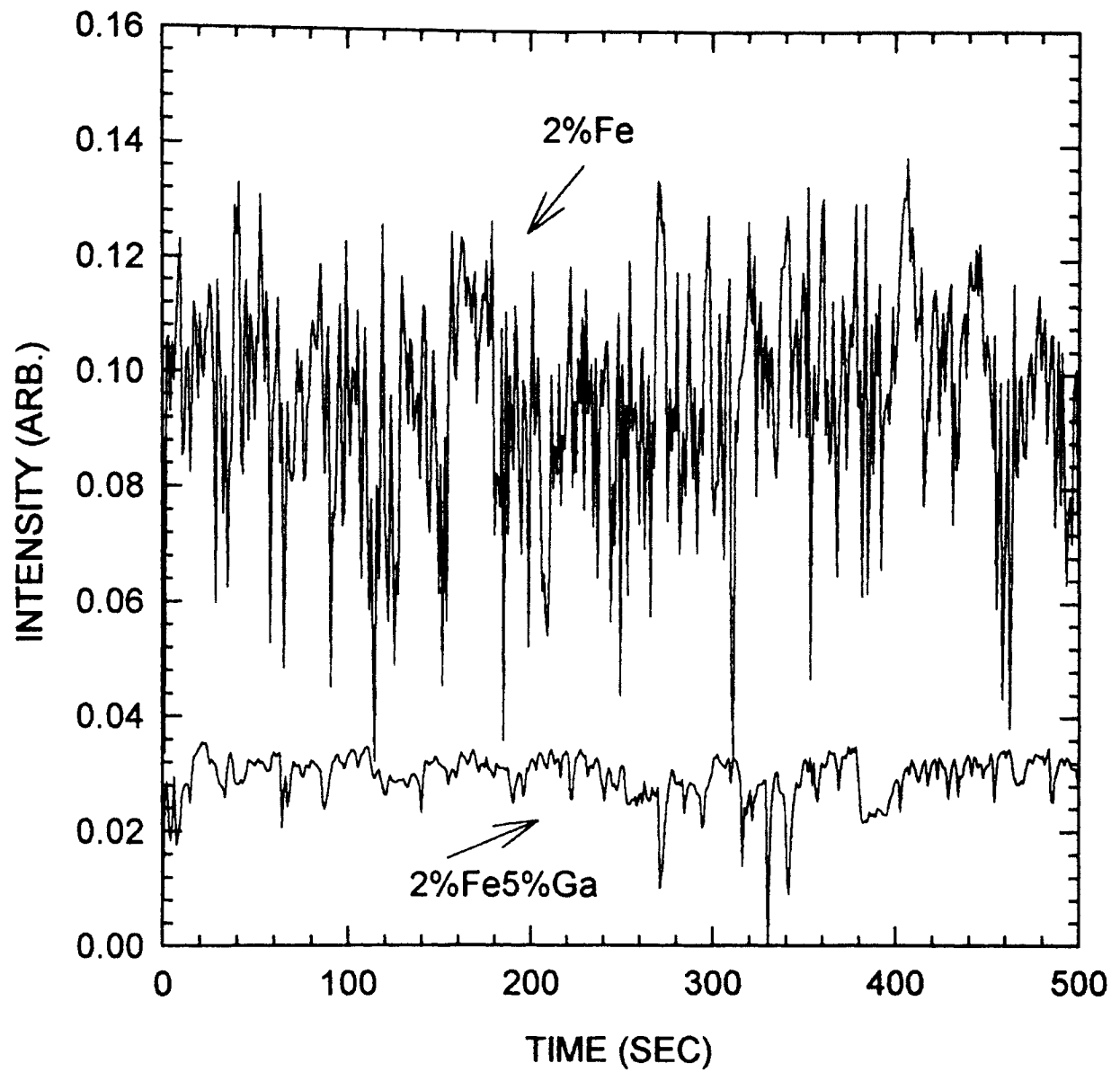
Figure 14. DVM real-time plot of refracted signal from BSO:2%Fe and BSO:2%Fe5%Ga
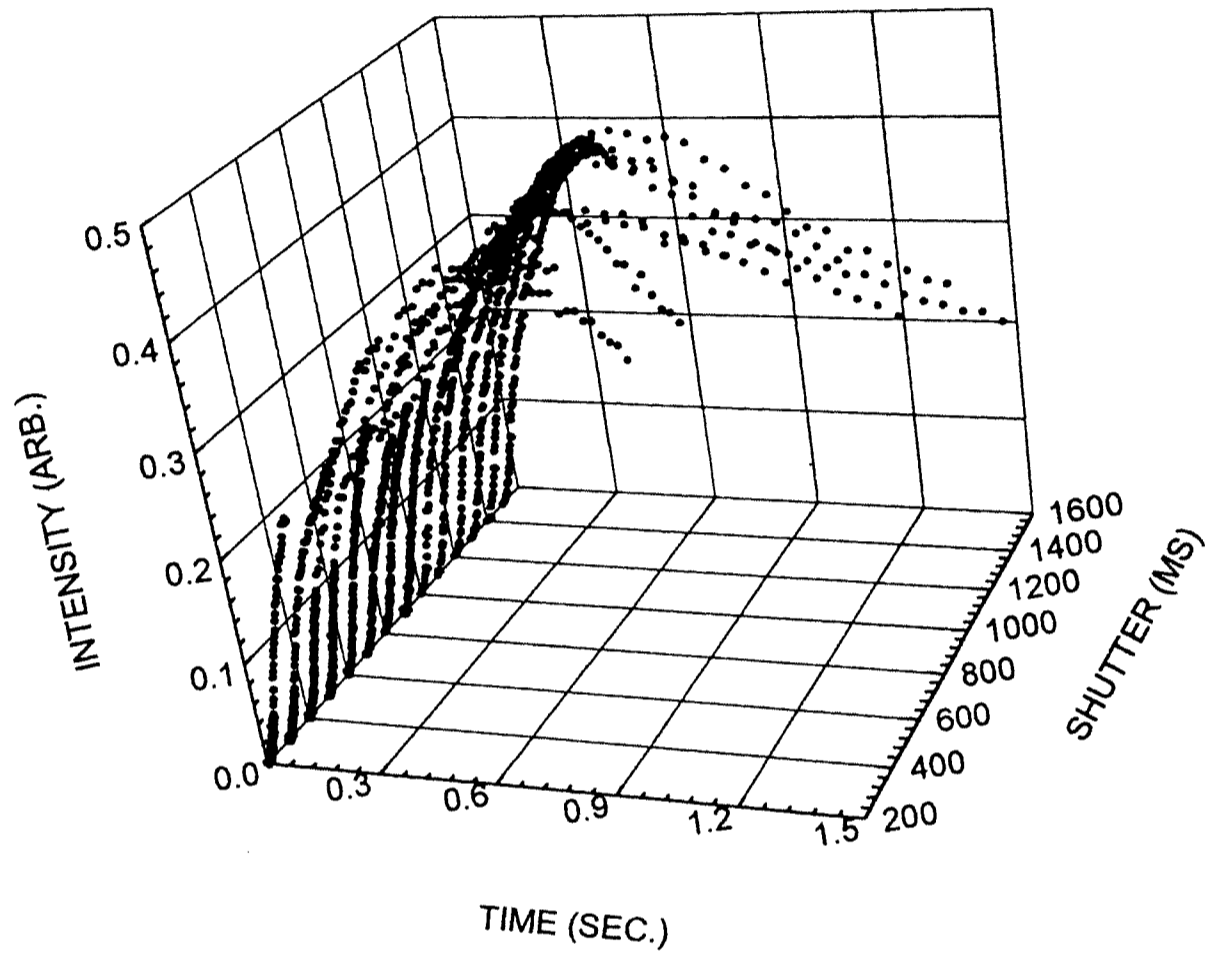
Figure 15. Shutter runs at room temperature for BSO:2%Fe
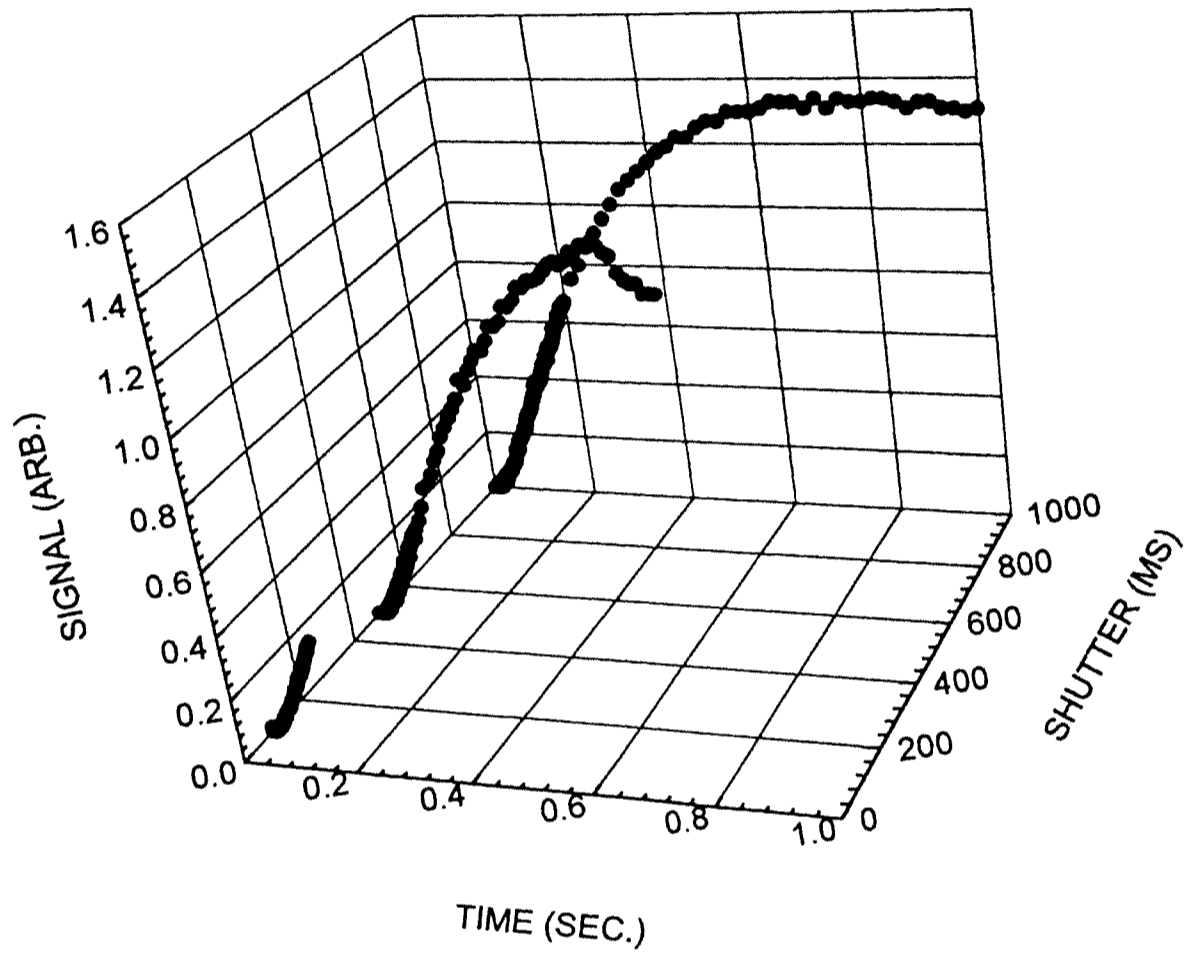
Figure 16. Shutter runs at room temperature for BSO:2%Fe5%Ga

Finally, to monitor the reproducibility of the data, the shutter was opened for 1000ms and consecutive readings were taken. This was done on both the BSO:Fe and the BSO:FeGa samples. The readings were taken immediately after each other at room temperature. Results are shown for Fe-doped in Fig. 17 and for FeGa-doped in Fig. 18. For the majority of trials, the results were close, however, in figure 18, there was one trial with a signal intensity that went off scale. It seems that signal intensity is largely dependent on the prior "immediate" history of the sample and possibly longer term effects not measured in this study
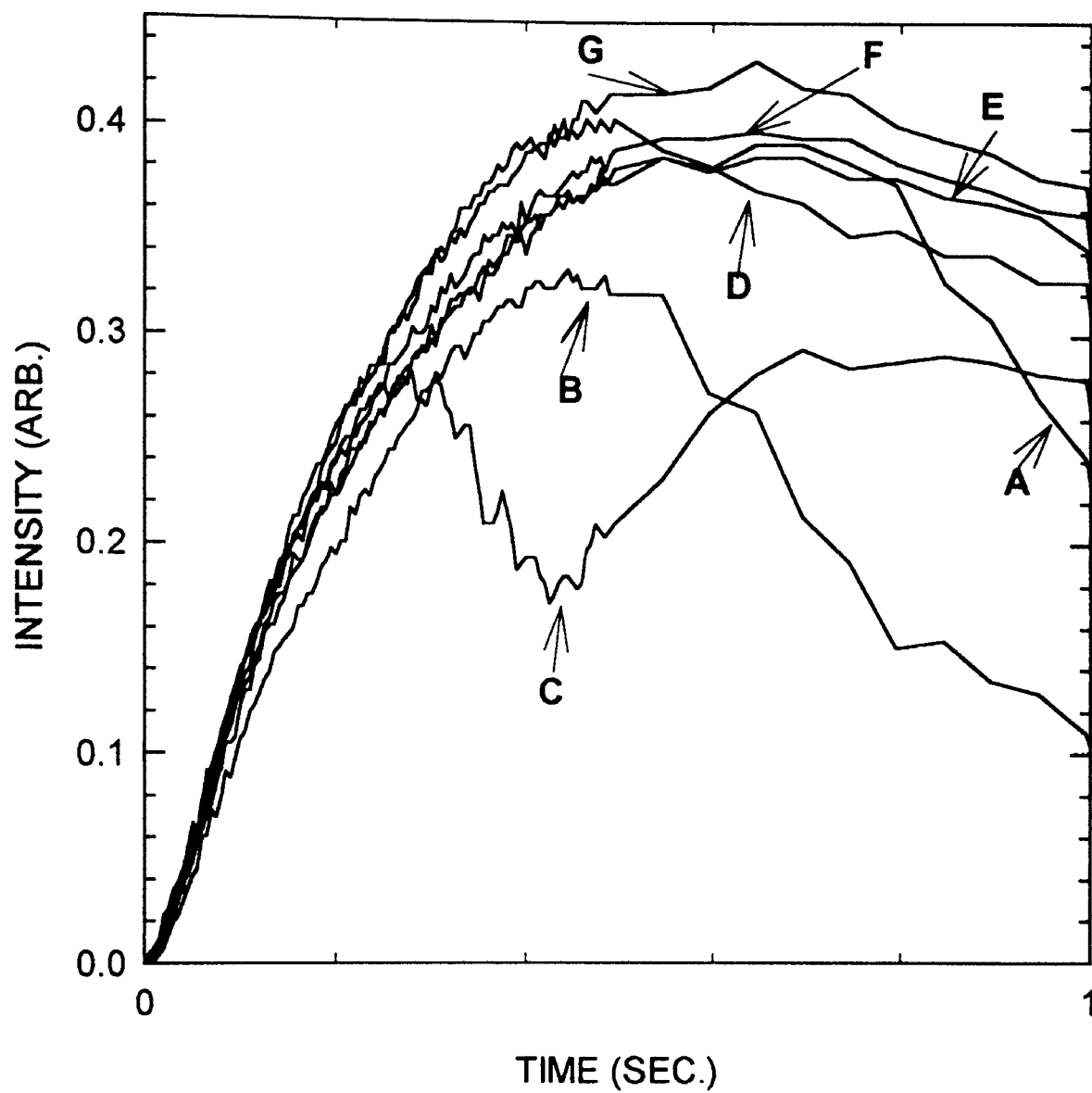
Figure 17.   1000ms Shutter runs for BSO:2%Fe

Figure 18. 1000ms Shutter runs for BSO:2%Fe5%Ga

CHAPTER 4

CONCLUSION

BSO:2%Fe and BSO:2%Fe5%Ga form photochromic absorption bands at low temperatures. These photochromic curves show interesting results at the absorption shoulders when compared with undoped BSO and Al-doped BSO. Undoped BSO, yellow in color, exhibits a strong absorption shoulder due to the presence of charge migrations from a deep donor to the conduction band. Al-doped BSO, however, has an absorption cut-off at 3.3 eV and is a nearly colorless crystal. The dopant occupies the Si site and acts as an acceptor t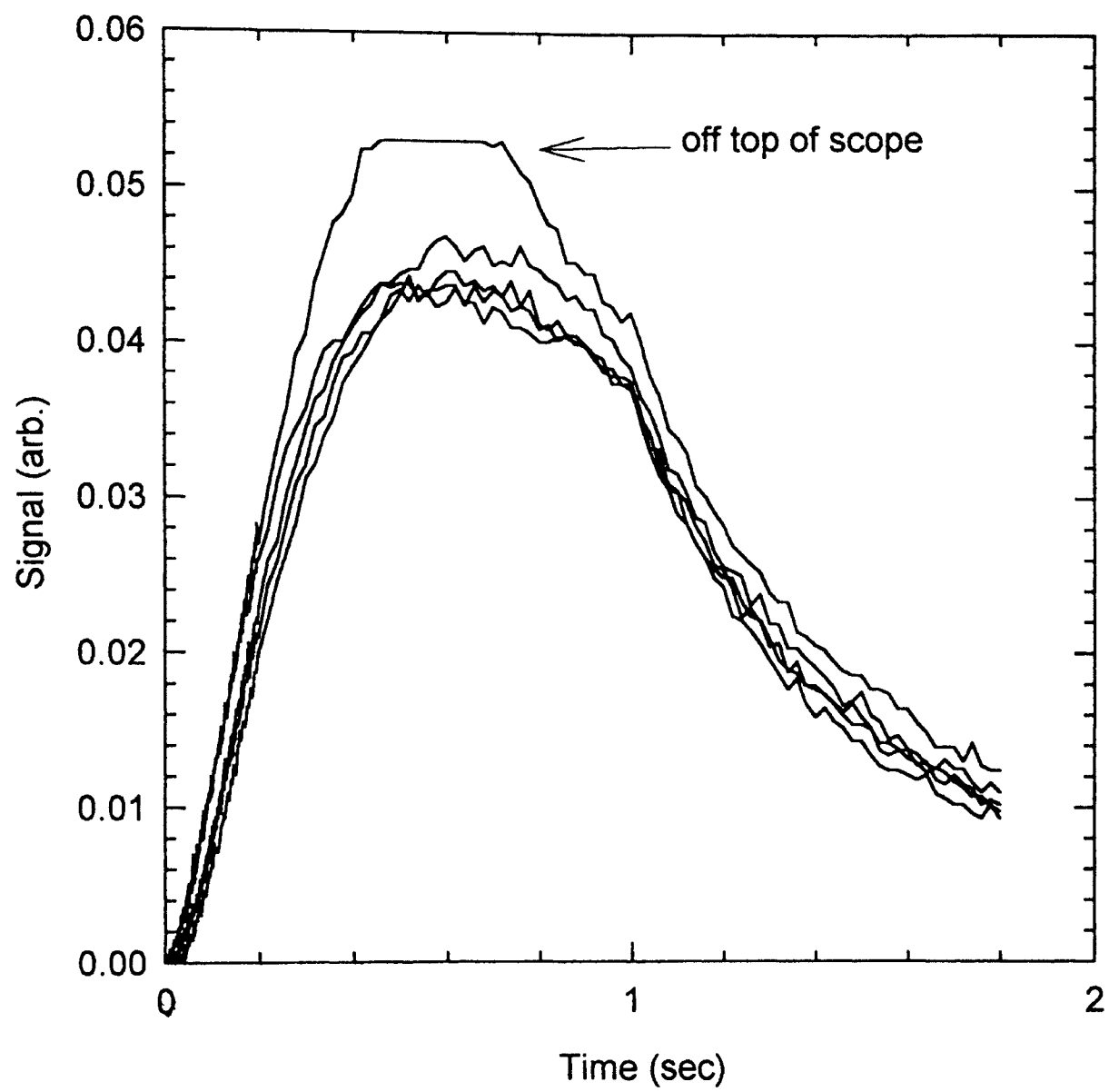o compensate the deep donor and thus removes the yellow coloration. Doping with Fe and with FeGa, also removes the yellow color, but the absorption cut-off is between 3 eV and 3.3 eV. It appears that the cut-off is due to an internal transition in the Fe, perhaps from the ground, spin 5/2 state, to the spin 3/2 excited state. Thermal excitation from the excited state at room temperature could then be responsible for the "winking" phenomena observed in the Fe-doped and FeGa-doped samples.

Photorefractive measurements show interesting trends in the temperature dependence of the Fe-doped and FeGa-doped samples. The mechanisms responsible for the change in the refracted signal strength at lower temperatures in the Fe-doped sample appear to be the same as those postulated for the absorption results. The FeGa-doped sample, however, shows unexpected behavior. The results show a mixing of two "competing" acceptor states located between 3 eV and 3.3 eV.

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APPENDIX A

DVM DATA ACQUISITION PROGRAM LISTING

```
10      CLEAR SCREEN
20      PRINT "THIS PROGRAM GRAPHS REALTIME DVM DATA"
30      DEG
40      ASSIGN @Isc TO 7
50      ASSIGN @Shutter TO 706    !PS PROG FOR SHUTTER TRIGGER
60      OPTION BASE 1
70      DIM T(1000),Sig(1000)
80      ON KEY 1 LABEL "AXES" GOSUB Grafaxes
90      ON KEY 2 LABEL "DATA" GOSUB Takedata
100     ON KEY 3 LABEL "Dump" GOSUB Dumpgraf
110     ON KEY 4 LABEL "STORE" GOSUB Storedata
120     KEY LABELS ON
130     INPUT "ENTER THE DRIVE & DIRECTORY AS:DR:\DIR\",Dr$
140     GOTO 140
150 Grafaxes:   !DRAWS THE AXES ON THE SCREEN
160     !SETUP THE SHUTTER
170     CLEAR SCREEN
180        INPUT "PLEASE TELL ME THE TIME INTERVAL FOR THE SHUTTER",Tshut
190        PRINT "REMEMBER TO ENTER THE SHUTTER TIME IN THE CONTROL BOX"
200        IMAGE  "THE SHUTTER TIME IS",4D.D
210        PRINT USING 200;Tshut
220        INPUT "ENTER THE TICK SPACING FOR THE TIME AXES",Ttick
230     !SET UP AND DRAW THE GRAPH FOR REALTIME PLOTTING
240        VIEWPORT 0,100*RATIO,0,100
250        WINDOW -.2*Tshut,1.05*Tshut,-15,15
260        CLIP 0,Tshut,-10,10
270        AXES Ttick,1,0,0
280        CLIP OFF
290        LORG 6
300        FOR Tm=0 TO Tshut STEP Ttick
310          MOVE Tm,-.5
320          LABEL Tm
330        NEXT Tm
340        MOVE Tshut/2,-2
350        LABEL "TIME"
360        LORG 8
370        FOR Sg=-10 TO 10 STEP 4
380          MOVE -.5,Sg
390          LABEL Sg
400        NEXT Sg
410        LDIR 90
420        MOVE -.05*Tshut,3
430        LABEL "SIGNAL"
440        LDIR 0
450     RETURN
460 Takedata:   !TAKES THE DATA
470        INPUT "ENTER THE CURRENT AMP GAIN IN VOLTS/AMP",Gain
480        Gain=1.E+4/Gain    !SCALE TO A MAX GAIN OF 1E4
490     !FIND Dvmavg
500        Dvmavg=0
510        FOR I=1 TO 20
520          ENTER 726;V
530          Dvmavg=V+Dvmavg
540        NEXT I
550        Avg=Dvmavg/20
560     !READ THE DVM AND PLOT THE DATA REALTIME
570        T0=TIMEDATE MOD 86400!SETS ZERO OF TIME SCALE
580          OUTPUT @Shutter USING "K";"2000"
590          WAIT .1
600          OUTPUT @Shutter USING "K";"2480"
610          MOVE 0,0
620          FOR I=1 TO 1000
630            ENTER 726;Sig(I)
640            Sig(I)=Gain*(Sig(I)-Avg)
650            T(I)=TIMEDATE MOD 86400-T0
```

```
660          DRAW T(I),(Sig(I)*100)
670          WAIT Tshut/1000
680       NEXT I
690       PRINT "I'M FINISHED, HIT ANOTHER KEY"
700       RETURN
710       PRINT "THE DATA IS IN THE COMPUTER, READY TO PRINT AND STORE"
720 Dumpgraf:        !
730                  CONFIGURE DUMP TO "HP-PCL"
740                  DUMP DEVICE IS 10,EXPANDED
750                  DUMP GRAPHICS #10
760                  PRINTER IS 10
770                  PRINT CHR$(12)
780                  PRINTER IS 1
790                  PRINT "I'M FINISHED, HIT ANOTHER KEY"
800                  RETURN
810 Storedata:    !STORE DATA ON DISC
820              INPUT "ENTER THE FILE NAME--F$",F$
830              !STORE THE DVM DATA WITH EXTENSION.DVM
840              ASSIGN @File TO *
850              CREATE Dr$&F$&".DVM",2*N+1
860              ASSIGN @File TO Dr$&F$&".DVM";FORMAT ON
870              DIM A$[50]
880              IMAGE  15A
890              OUTPUT @File USING 880;"DVMDATA"
900              IMAGE  3D.2D,",",SD.DDE
910              FOR I=1 TO 500
920              OUTPUT @File USING 900;T(I),Sig(I)
930              NEXT I
940              ASSIGN @File TO *
950              PRINT "I'M FINISHED, HIT ANOTHER KEY"
960              RETURN
970       END
```

VITA

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Master of Science

Thesis: PHOTOCHROMIC AND PHOTOREFRACTIVE EFFECTS IN
BISMUTH SILICON OXIDE DOPED WITH IRON AND BISMUTH
SILICON OXIDE DOPED WITH IRON AND GALLIUM

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