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PANCHROMATIC AND SPECTRAL CHARACTERIZATION OF Cu CONTAMINATED SEMI-INSULATING GaAs

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Abstract

Panchromatic (integral) and spectrally resolved cathodoluminescence characterization was used to investigate the near surface gettering properties of Cu in liquid-encapsulated, Czochralski-grown, undoped semi-insulating (SI) GaAs. Samples from two sources were investigated to determine if gettering treatments applied to GaAs result in improvements in uniformity similar to those observed in gettered Si. Before Cu contamination, typical cellular structure is observed for all samples. Experimentally, it is found that the panchromatic CL images change significantly after Cu doping and subsequent gettering processing for all samples. A contrast reversal is generally observed after Cu contamination. After gettering, the image of the samples from one source remained reversed whereas the image of samples from the other source showed a second contrast reversal. Typically, both samples exhibit bright regions after gettering which closely correspond to the dislocation structure. More detailed spectrally resolved CL indicates that Cu luminescence correlates well in most cases with the band edge emission. In only a few cases were discernible differences noted. It is concluded that Cu is observed in locations from which nonradiative recombination centers have been effectively removed.

Key Words: Spectrally Resolved Cathodoluminescence, Panchromatic Cathodoluminescence, Cu Contaminated, SI-GaAs, CL Contrast Reversal, Gettering.

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Introduction

Liquid encapsulated Czochralski (LEC) semi-insulating (SI) GaAs is very attractive for a large variety of applications, one of those being high speed integrated circuits which are inherently radiation hard. Unfortunately, these circuits are found in only the most high performance markets where cost is a secondary issue. One reason for lack of more widespread application is the well known fact that GaAs shows significant electrical nonuniformities across a wafer, even over distances of several hundred microns. The resultant characteristics of metal semiconductor field effect transistors (MESFETs) fabricated on SI GaAs show non-uniformities in threshold voltage, resistivity, leakage current, mobility, and transconductance [14,21]. Correlation with the defect structure (dislocation network) is commonly observed. Gettering techniques provide one alternative for better uniformity.

Gettering is simply any technique which improves electrical characteristics of the active area of the substrate through migration of defect species. In general, the gettering process involves the removal of native defects and impurities or the removal of their electrical effects from the active device regions. The resulting crystalline structure is purer and exhibits improved properties.

Defect patterns in as-grown LEC GaAs have been studied using cathodoluminescence [2,4,8,12,13,16,18]. Dislocations generally appear as dark spots about 5 μm in diameter. In many cases, around these dark spots there are very bright rings about 20 μm in diameter. This pattern is known as a 'dot and halo' image. The dark spots have been interpreted as locations of high nonradiative recombination centers. The light regions have been interpreted as denuded zones. The defects which lower the non-radiative recombination lifetime have been removed from this region. It is thought that the impurities in the 20 μm circle are gettered to the dislocations leaving the surrounding material clean. Dislocations introduce an energy attractor for impurities causing the migration of impurities from the bulk material to the dislocation [9]. Once the impurities have been drawn to the dislocation, the impurities can move along the dislocation very easily in a pipeline type of motion if additional attractors exist, such as the stress fields associated with surface damage.

In one experiment, GaAs material was heat treated to determine if the CL image would change [18]. When the sample was annealed at 1050°C for 24 hours and rapidly quenched, the CL image did not show the as-grown "dot and halo" image which leads to the conclusion that there is a temperature/time effect associated with the gettering of impurities. If the sample is then annealed and slowly cooled, similar to as-grown material, the "dot and halo" images appear

again. It was determined that the impurities redistribute themselves only for an anneal temperature of over 750°C. Additionally, post growth heat treatments appear to improve the uniformity of the GaAs [16].

The distribution of dislocations across a wafer is observed to be nonuniform. On the wafer scale, the dislocations typically show a four-fold symmetry with regions of high dislocation density separating each quadrant [7]. A radial symmetry is also observed with high dislocation densities in the center and at the edges. This is typical, but not always measured since other distributions are sometimes observed [1]. On a microscopic scale, the dislocations formed during growth form a cellular structure with dimensions as large as several hundred microns. These dislocations are found in the walls of the cellular regions while the interiors are found to be relatively dislocation free. Since defects and impurities interact with the strain fields of the dislocations, it is not surprising that electrical nonuniformities exist on the wafer scale and the chip scale. Hence there is interest in gettering studies to improve uniformity.

The cellular structure is easily observed by CL. The cell walls appear bright, with dark pinpoints corresponding to dislocations. The cell interiors are in general dark compared with the cell walls. Etching studies conducted in conjunction with CL have verified this identification. One such study using A/B etchant, which is sensitive to the Fermi level, showed close correlation with the contrast pattern described above [3]. Similarly, a one-to-one correlation between the dark spots in CL images and KOH etch pits within cell walls has also been established [16].

Resistivity maps of semi-insulating GaAs have been determined and correlated to CL images using charging microscopy [20]. Since the sample is semi-insulating, it is more likely to be affected by charging effects with local conductivity (resistivity) determining the amount of charging. A secondary electron image which corresponds to the relative charge distribution (bright in negative regions, dark in positive areas) due to current flow through the sample results in a resistivity map. Again, inverse correlation with CL is observed.

Cathodoluminescence has several advantages as a characterization technique. It provides microanalysis with resolution on the order of 1 μm which is typically better than most PL implementations. CL can be used to characterize most any luminescent sample regardless of the resistivity of the sample unlike the electron beam induced current (EBIC) measurement which is difficult for high resistivity samples. The most important features of CL are that it is non-contacting and non-destructive. This allows CL to be used as a process monitor where samples can be measured and replaced in the process without ill effects.

Materials and Methods

CL was used to characterize samples to study the gettering properties of Cu in SI-GaAs. Samples were intentionally contaminated with Cu since it is an impurity known to cause problems in GaAs devices [19]. Contamination was necessary to raise the concentration to a level such that the effects of gettering could be clearly observed.

The luminescence signature associated with the substitutional copper defect (Cu_{Ga}) is found at T = 5 K at 1.36 eV (911 nm) with associated phonon replicas at lower energy. This was used to identify the presence of Cu in both the PL and CL measurements in this study. Other impurities are also known to exhibit luminescence near this energy. Since no emission near this energy was observed in the starting material and the 1.36 eV peak was only observed after Cu contamination, it was assumed to be due to Cu alone.

Samples were selected from double-side polished, 500 μm thick undoped SI-GaAs. Due to the typical "W" pattern of dislocations and other electrical and chemical properties, sample sets were composed of 0.5 square-inch samples selected from mirror locations around the wafer [14]. This selection helped to eliminate variability of results due to the micro-structure properties of SI-GaAs.

The sample sets were further split into three groups. One group was held as a control. The second group was intentionally contaminated by evaporating a layer of Cu on the surface of the samples. The samples were then annealed for 12 hours at 550°C. Due to the high diffusivity of Cu, it distributes throughout the bulk of the material quickly. Transient effects due to multiple diffusion configurations with native defects were not expected [17]. The third group was also contaminated with Cu and further submitted to a gettering step. The gettering step consisted of uniformly damaging the back surface with 30 μm or 45 μm diamond grit for 2 minutes. The samples were then heat treated for another 12 hours at 550°C.

The anneal temperature of T = 550°C was determined by a combination of factors. At this temperature, the diffusivity of Cu is sufficient to ensure uniform distribution of the contaminant in the bulk. Additionally, the temperature is less than 657°C, the temperature above which surface decomposition becomes a significant concern [11]. Hence, capping was not used to protect the surface.

The activation energy for these experiments was supplied by thermal heating, carried out using a "leaky tube" diffusion furnace setup for annealing. A schematic of the system is shown in Figure 1 and its operation has been described by Roedel, *et al.* [15]. This method allows the annealing of samples in a stagnant atmosphere which approximates the sealed ampule method. Although As overpressure is not utilized with this procedure, this was not a problem because of the low temperatures utilized in this study.

For annealing, the following procedure was used. The samples were degreased and placed on a fused quartz boat on edge and loaded into the mouth of the furnace. The system was then purged through the purge line in the inner liner with He for one hour to remove any residual gases that were present in the system. The samples were then slowly moved into the center of the furnace for the anneal. The samples were annealed for 12 hours at 550°C. At the end of the cycle, the samples were slowly pulled from the hot portion of the furnace into the mouth of the furnace. The samples were allowed to cool for at least one hour after which the furnace was opened and the samples were removed.

The samples were first characterized by panchromatic CL with a S-1 photomultiplier tube (PMT) using a JEOL 840 scanning electron microscope (SEM). (The primary electron beam current was typically 2×10^{-7} A, and the accelerating voltage was 30 kV.) Clear changes were observed between the groups of samples. Unfortunately, a clear indication of the source of the change could not be determined from this information alone. The changes could not be directly attributed to the Cu contamination alone.

A spectrally resolved CL system was used to help track the different species to determine the source of the observed changes in panchromatic CL. A schematic of the system is shown in Figure 2. The system consisted of both commercial and specially designed components. The JEOL 840 SEM was used with a modified Oxford Instruments CL cold stage capable of near T = 15 K operation. Samples were mounted using a thin layer of silver paste. The generated light was directed to an Instruments SA HR320 spectrometer (1200 g/mm) and then to the appropriate detector. All of the optical components were made from CaF_2. The band edge CL was directed to a Hamamatsu R928 PMT and the output was fed to the SEM.

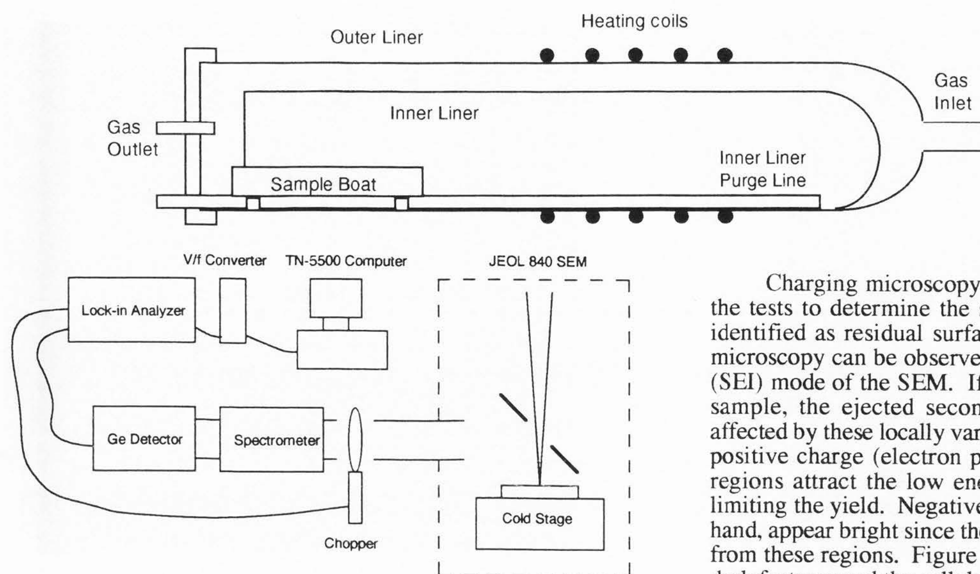


Figure 1. Leaky-tube diffusion furnace schematic.

Figure 2. Spectrally resolved cathodoluminescence experimental schematic.

For longer wavelength radiation, the chopped CL signal was detected with a liquid nitrogen cooled North Coast Scientific Ge detector. The Ge detector is known to have spiking problems due to extraneous high energy radiation (muons). This introduced significant noise into the captured images since no electronic filter circuitry was used. The AC output of the Ge detector was measured with a Princeton Applied Research model 5301 lock-in analyzer. The lock-in output voltage was then fed to a custom designed voltage-to-frequency converter then to a Tracor-Northern TN-5500 computer. Time for acquisition of Cu images ranged from 15 min. to over 1 hour.

Before spectral CL analysis, all samples were characterized using photoluminescence (PL). This allowed for the rapid determination of the emission spectrum expected from each sample. Several samples were rejected based upon the strengths of the PL signals. Those with strong Cu signature were characterized using the spectral CL system.

Experimental Results

Panchromatic CL was performed on semi-insulating samples from two vendors. Both materials showed the expected cellular dislocation structure. However, this was the only similarity between the samples (Figure 3). Several wafers were grown at Arizona State University (ASU) and others at Litton Airtron. The ASU material showed thin, well defined cellular walls with a high contrast between the cell walls and the cell interiors. The interiors of the cells were in general dark with dimensions on the order of 100 μm. On the other hand, the Litton Airtron material showed very broad diffuse cellular walls which surround relatively small cell interiors. This was the first indication that internal properties differed between the two materials even though they are both semi-insulating.

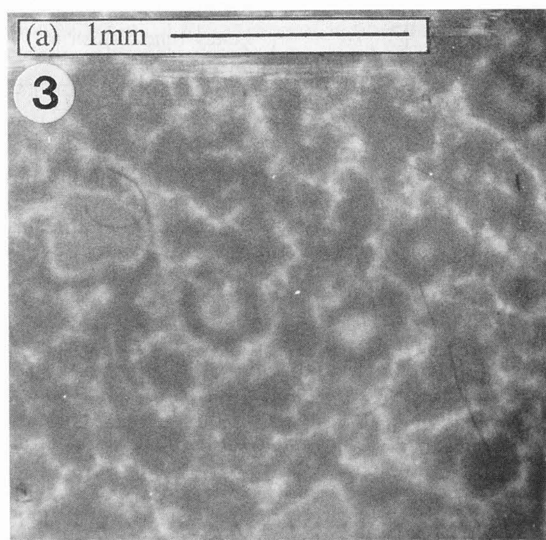
Dislocations were relatively easy to observe with CL. They appear within the bright cellular walls as dark spots. This is clearly seen for ASU Ware SI-GaAs in Figure 4. The dark spot features were only observed in the bright cellular walls arranged generally along a single line along the cell walls. Similar features were observed for Litton Airtron samples, the only difference being that the walls were wider and many dark features were found across the wall.

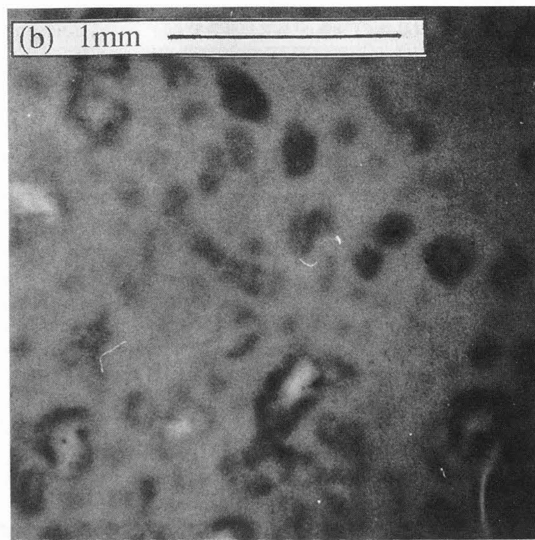
Charging microscopy was incidentally observed during the tests to determine the source of the dark linear features identified as residual surface damage (Figure 5). Charging microscopy can be observed in the secondary electron image (SEI) mode of the SEM. If a voltage profile is present in the sample, the ejected secondary electrons may be strongly affected by these locally varying electric fields. Those areas of positive charge (electron poor) will appear dark since these regions attract the low energy secondary electrons thereby limiting the yield. Negative areas (electron rich), on the other hand, appear bright since the low energy electrons are repelled from these regions. Figure 5 shows a correlation between the dark features and the cellular structure observed with charging microscopy.

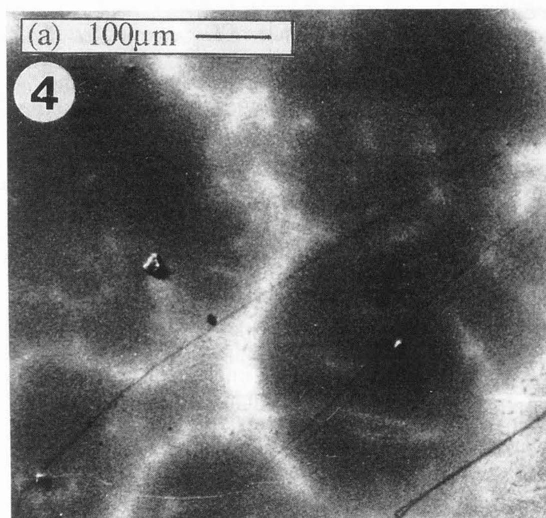
Room temperature panchromatic CL images obtained for gettered Litton Airtron material doped with Cu are shown in Figure 6. SEM CL showed significant changes among the SI-GaAs, the Cu contaminated, and the gettered samples. After contamination, the cellular structure was still observed, but the dark interior regions were now brighter than the cell walls. This indicates that the ratio of the radiative lifetime to the nonradiative lifetime has changed. Some dark features remain while bright clustering in the dislocation walls emerges. However, after gettering, the dark pinpoints of the dislocations in the walls are not observed while more bright clustering appears. The most significant change is the contrast reversal and the emergence of the bright cluster regions within the cellular walls. The difference in brightness (relative contrast) between the cell walls and the cell interiors also decreased indicating a trend toward more uniform material.

A gettering CL sequence consisting of as-received, contaminated, and gettered material is shown for ASU material in Figure 7. There are some differences from the characteristics observed for Litton Airtron material. The starting material has bright cellular walls that are relatively thin, surrounding darker cell interiors. After Cu contamination, the cell interiors become brighter than the cell walls as was observed with the Litton Airtron material. A significant difference was observed in the walls. Bright pinpoints scattered along the walls begin to emerge at this point. Once again, after gettering, the contrast between the cell interiors and the cell walls is reduced. The bright pinpoints in the walls themselves become more prominent and more prevalent. The CL contrast reversal of the dislocations follows that of the Litton Airtron material. On the other hand, contrast reversal occurs initially at the cell interiors for the ASU material after Cu contamination, but the image is again reversed after gettering. Unfortunately, the final reversal does not return the CL exactly to that of the pre-treated material due to the fact that the cell walls appear differently from the pre-treated samples. Thus, the conclusion that the gettering treatment is able to restore the material to the pre-contaminated conditions can not be made directly.

A closer look at the bright pinpoints in the ASU material shows interesting characteristics. Both bright and dark pinpoints associated with the cellular network are observed (Figure 8). Differences are seen in Figure 8 between the Cu-doped and gettered samples. There is a larger number of both dark and bright features in the cell walls. In fact, there are







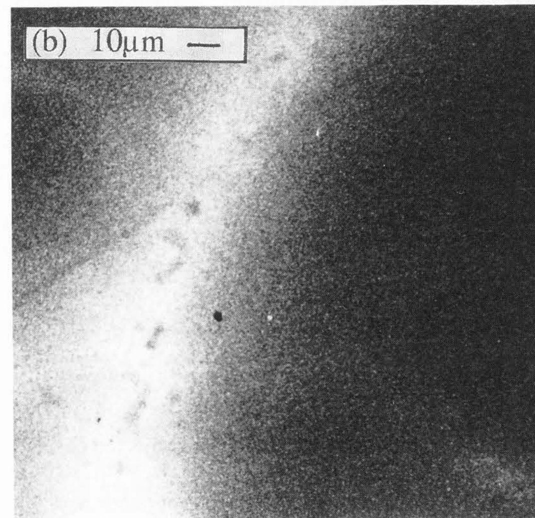


Figure 3. Cellular structure observed using room temperature panchromatic CL for SI-GaAs: a) ASU Ware and b) Litton Airtron samples.

Figure 4. Room temperature panchromatic CL of dislocations located in cellular walls: a) low magnification and b) higher magnification.

more dark features and they are less associated with the bright features. The formation of both bright and dark features are enhanced after gettering treatments.

The observations of panchromatic CL are interesting, but no direct conclusions relating to Cu can be drawn. Spectrally-resolved CL was used to investigate a select number of samples after PL characterization. The three bands specifically imaged included the band edge exitonic peak (820 nm), the carbon peak (830 nm), and the Cu peak (911 nm). A representative spectrum is shown in Figure 9. It is clear that the Cu signal was very weak, requiring long data acquisition times.

Lower magnification images were obtained for the Cu contaminated Litton-Airtron material (Figure 10). The same

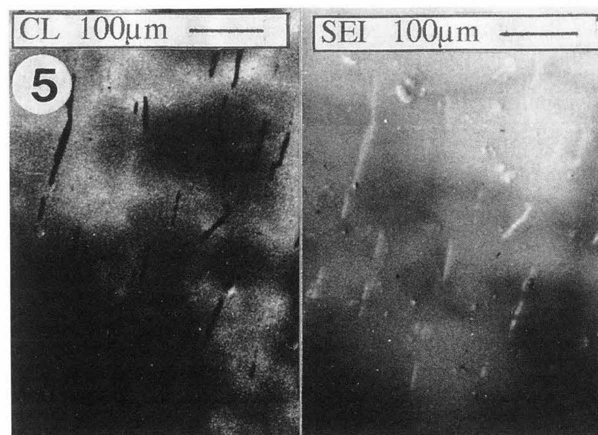
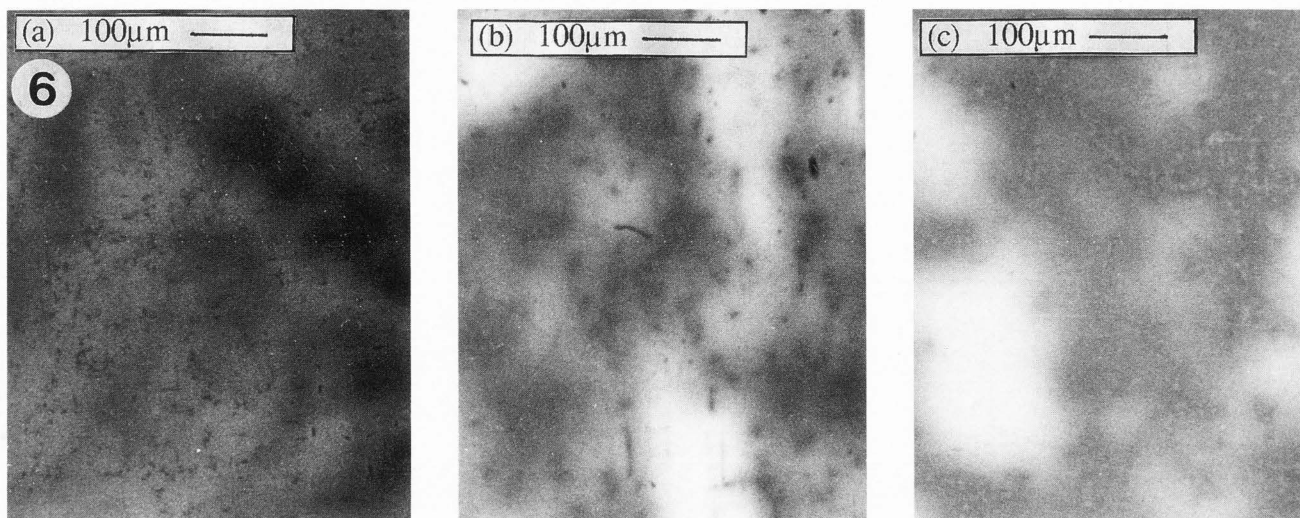


Figure 5. Charging microscopy example correlated to CL images for SI-GaAs.



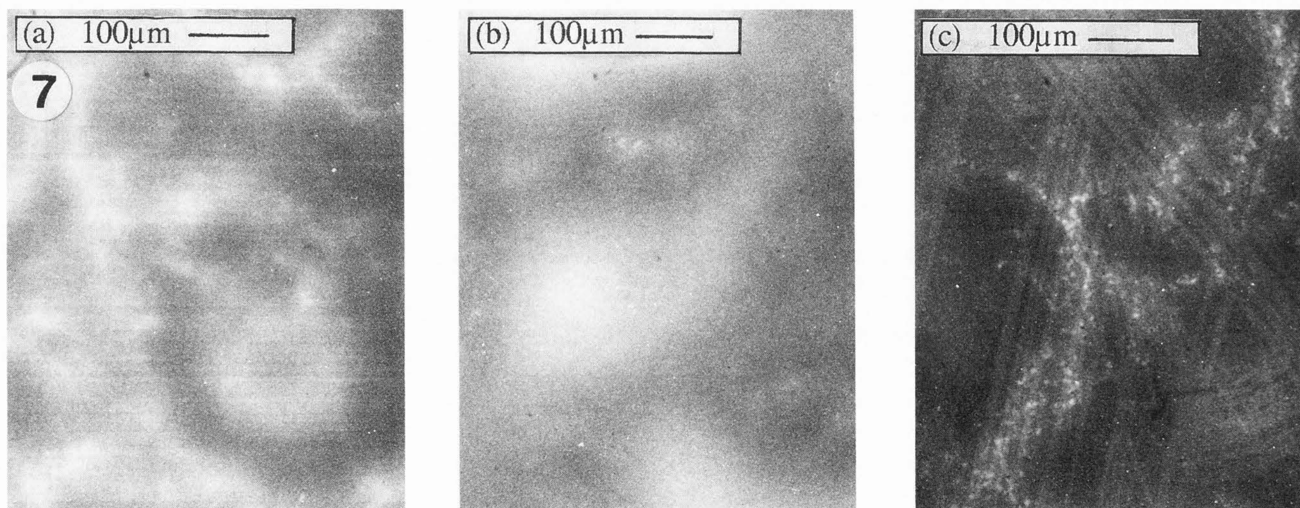


Figure 6. SEM room temperature panchromatic CL of Litton Airtron Material doped using Cu evaporation a) SI-GaAs b) GaAs:Cu c) 30μm gettered.

Figure 7. SEM room temperature panchromatic CL of ASU Material a) SI-GaAs b) GaAs:Cu c) 30μm gettered.

general features observed in the room temperature panchromatic CL images emerge. This is expected in light of the analysis of the earlier photomicrographs. Of more interest are the carbon and copper images. The carbon image is well correlated to the band-edge excitonic image but shows less contrast. The copper image was the most surprising in its high correlation with the band edge image. It was expected that the Cu would segregate and show different distributions.

Higher magnification images were obtained for the Cu-contaminated ASU material (Figure 11). Again, the cellular structure is seen with several distinct dark features (dislocations). The carbon image is also well correlated with the band-edge signature (not shown). The copper image again shows striking correlation with the band-edge image. If Cu were responsible for the observed room temperature CL contrast changes, less correlation and even inverse correlation would be expected.

Discussion

Charging microscopy may be used to help interpret the CL image since it correlates well with the cellular structure in the CL image. Those areas that appear bright are electron rich (negatively charged) indicating higher relative resistivity since the deposited charge from the electron beam is not dissipated as rapidly as those areas which are dark. Electrically, this means that the effective doping density $|N_A - N_D|$ is less in the bright regions. Charging microscopy and CL show an inverse correlation indicating that the areas that are more optically active have a higher effective doping density. This does not necessarily mean that the concentration of the species of interest (i.e. Cu) is changing. It could also indicate that other defects, intrinsic or extrinsic could be the cause of the nonuniformity.

Contrast reversal has been previously reported [6,9]. However, one of the investigations indicated that a high temperature anneal ($T > 800°C$) must be used for contrast reversal to occur [9]. This is contrary to our observations. We see contrast reversal at significantly lower temperatures. A possible explanation is that during growth, fast diffusing

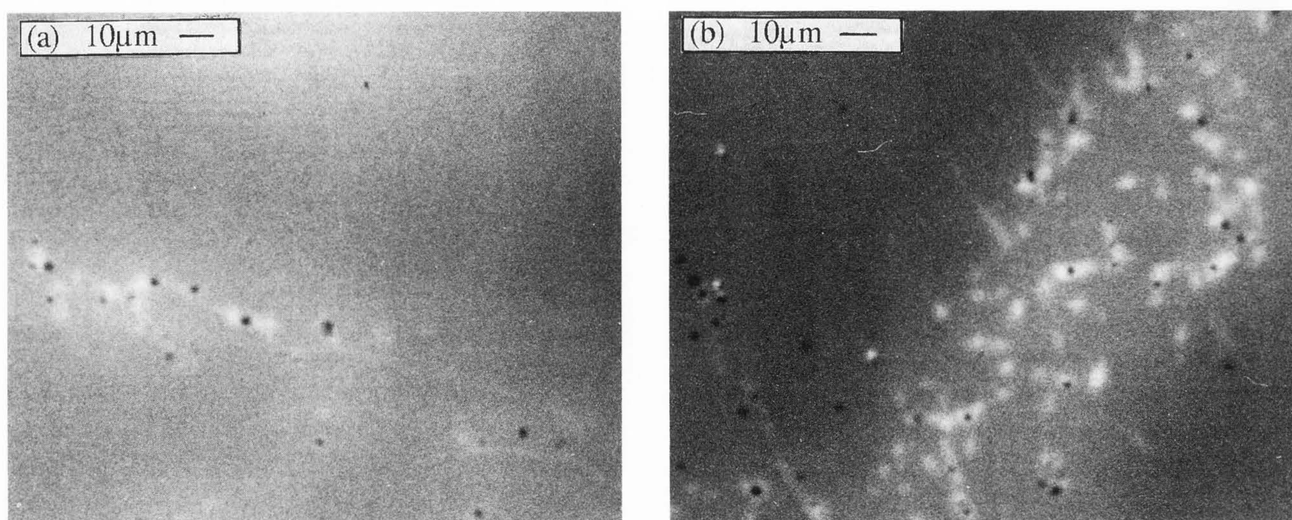


Figure 8. Additional CL features observed in material from ASU: a) Cu doped and b) gettered.

impurities are attracted to and saturate the dislocations. Once a dislocation becomes saturated, attraction of additional point defects is inhibited. This occurs before complete gettering from the cell interiors has occurred; hence the dark cell interiors of the starting material. Once the surface of the sample is damaged, point defects along saturated dislocations are attracted by new point defect-free stressed regions. This reduces defect saturation along dislocations and again fast diffusing point defects are attracted from cell interiors. After sufficient heat treatment, the cell interiors become essentially denuded of fast diffusing point defects resulting in bright cell interiors.

Consider the bright and dark features noted in the ASU material (Figure 8). Contrast reversal is observed which would account for the bright pinpoints associated with the dislocations themselves. It is known that if the carrier concentration is less than $10^{18}/cm^3$, CL intensity increases monotonically with carrier concentration. Thus, it has been proposed that the normally dark dislocation features become bright due to the increased free carrier concentration associated with the gettering of electrically active impurities [5]. The local radiative recombination becomes greater than the nonradiative recombination attributed to dislocations. A troubling feature of this explanation is that the gettered species must not contribute significantly to nonradiative recombination. Rather, we wish to getter these point defects which decrease the CL efficiency.

The dark features, however, introduce a complication. Many of the dark features are surrounded by bright regions. This is typical of dot-halo features which takes the above explanation a step further. The dislocation, having attracted a high concentration of defects, is so nonradiative that the center appears dark with the surrounding regions brightened.

The proposed explanation is appealing for many of the features in Figure 8. Unfortunately, there are several dark features not associated with bright rings. This indicates the possibility that another gettering path is in operation which does not involve the same species. The dark feature may be due to either a different type of dislocation or different defect such as a large inclusion. These features do not show the typical gettering ring indicating that the surrounding material is not cleansed of the species that inhibit luminescence. The feature, however, is electrically active, providing a path for

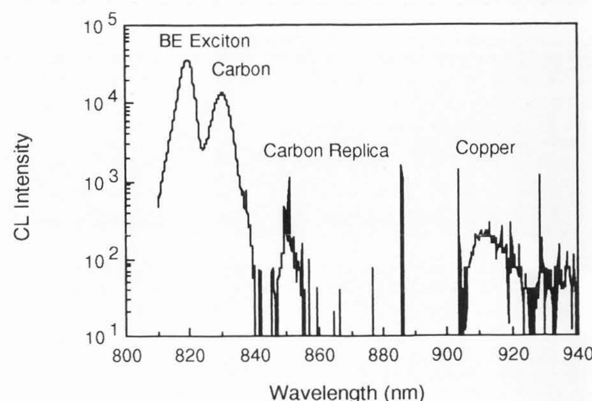
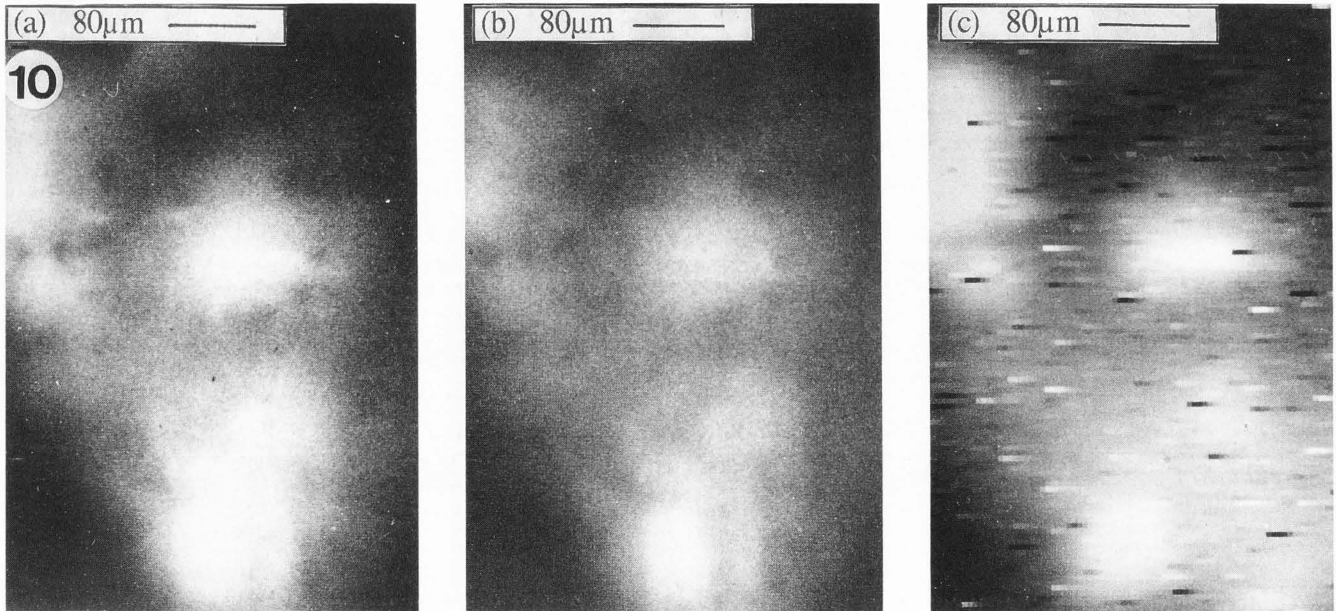


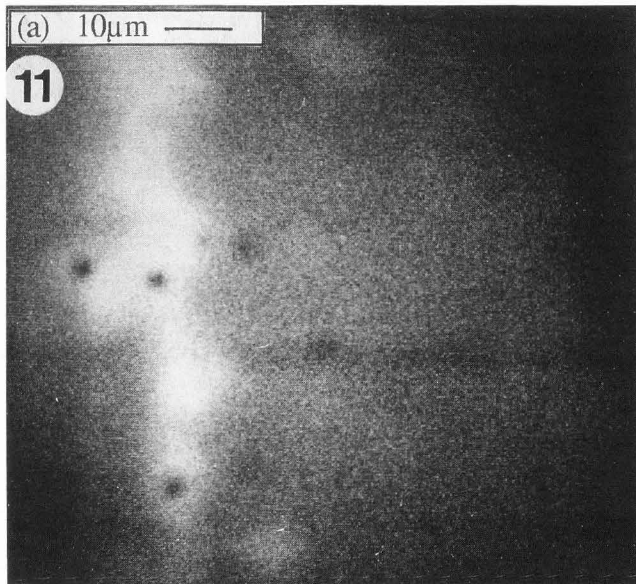
Figure 9. Example low temperature luminescent spectrum from Cu doped sample.

nonradiative recombination. Lifetime profiles around this type of feature have not been determined. These types of features have been reported for undoped GaAs in the analysis of simple heat treatments at 850°C [10].

The results of spectrally resolved CL provide several insights into the changes observed in the panchromatic images. The clear segregation effect of gettering which was expected to be observed for Cu is lacking. It is clear from the high degree of correlation that the processes that affect the luminescent images are not directly attributable to copper. In light of charging microscopy, panchromatic CL, and spectrally resolved CL, the changes are most likely due to the redistribution of other defects that are non-radiative in nature (or not observed with our detectors).

Additional electrical measurements were made on several samples including photo-induced transient spectroscopy, thermally stimulated current, and temperature dependent photoconductivity. The latter measurement is a spectrally resolved measurement which is reported elsewhere [22]. Interestingly, these measurements, though lacking in spatial resolution, do observe gettering effects in some of the samples. This indicates that complex interactions are responsible for the observed CL contrast changes. The question of why no differences were observed in the spectral CL images is not definitively answered by these measurements.





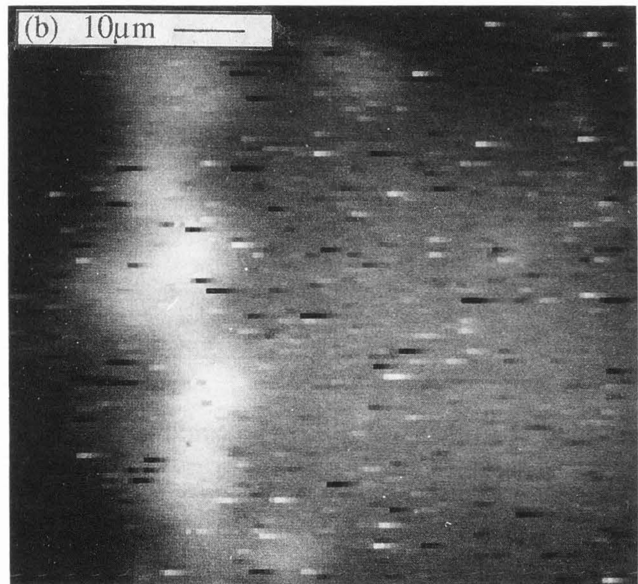


Figure 10. Low temperature spectrally resolved cathodoluminescence of Litton Airtron material for (a) band edge exciton, (b) carbon peak and (c) Cu peak.

Figure 11. Low temperature spectrally resolved cathodoluminescence of ASU material for (a) band edge exciton and (b) Cu peak.

Conclusions

The SEM panchromatic CL technique is useful in tracking spatial changes in the radiative quantum efficiency with gettering treatments. It is clear that the treatments do affect the samples and some homogenization is observed indicating the possibility of improved electrical behavior. Unfortunately, the spatial location of Cu cannot be unambiguously identified. Hence, the specific interaction of dislocations and external gettering treatments cannot be determined. This can only be done using the spectrally resolved CL technique.

Use of the spectrally resolved CL technique on these specific samples to observe the Cu substitutional defect indicates that the changes cannot be assigned to the introduction and subsequent motion of copper. The changes are more likely due to the redistribution of other defects that are not observed through luminescence. These include non-radiative defects and other radiative deep level defects that are not detected with our instruments.

Even though panchromatic CL cannot specifically track a given emission line, this technique should not be discarded.

Since LEC GaAs is very nonuniform electrically and optically, the spatial information provided by this technique is very valuable. Any change in the uniformity is useful in attempting to gain an understanding of impurity and defect motion due to specific treatments. The changes observed here indicate that impurities and defects are indeed moving and altering the uniformity.

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Discussion with Reviewers

A. Jakubowicz: The authors study Cu-contaminated GaAs. They conclude that the observed changes cannot be assigned to the introduction and subsequent motion of copper. They say in their "Conclusions" that the changes are more likely due to the redistribution of other defects. If so, then this is in some contradiction to an earlier statement (in "Materials and Methods") saying: "contamination was necessary to raise the concentration to a level such that the effects of gettering could be clearly observed." If other defects are responsible for the observed contrast variations, then the question arises, why was Cu contamination necessary to see gettering effects. Were these "other defects" created during the process of contamination with copper; were they generated during the subsequent gettering procedure of the Cu-contaminated samples; or lastly is the presence of copper needed to obtain a redistribution of other defects (originally present in the samples)? One group of samples was held by the authors as a control, but it is not clear if samples from this group were treated similarly to those contaminated by copper. Such a comparative experiment might be helpful in the interpretation of the authors' results.

Authors: We are unable to draw the conclusion that the changes in the luminescence is due to Cu alone from the data presented here. Cu contamination is required to introduce or raise the level of an extrinsic impurity to be observed using luminescence. We believe that attempting to determine the gettering behavior of an extrinsic impurity is less difficult than interpreting the interactions between the native defects which do not all have luminescence signatures. Other techniques (temperature dependent photoconductivity, FTIR, and thermally stimulated current) were used to characterize this material which did indicate a gettering of the Cu. With the introduction of Cu, additional defects are expected to be produced (complexes with Cu), however, the native defects already present in the material (EL2, EL0, etc.) are still the dominant group due to their high concentration. Redistribution of these defects during annealing is not

unexpected since the equilibrium distribution was reached during whole ingot annealing, before the wafers were cut. After the wafers are cut, new gettering sites are generated at the surfaces. At this time, the control set of samples have not been subjected to the same heat cycles of the Cu contaminated samples.

J. F. Bresse: As suggested by your CL images on the Litton Airtron sample, the interior of the cell is darker than the cell walls which are very wide on these samples. Figures 6b and 6c show more an increase of contrast than a contrast reversal. Figure 6b seems to show a migration of dislocations to the cell walls. Can you explain and comment?
Authors: The dark features in the images are the grown-in dislocations which are pinned and not known to migrate during subsequent annealing. Figure 6a shows the dark features identified as dislocations in the bright walls surrounding dislocation free dark cell interiors. Further, the dark features in Figure 6b are the dislocations of the walls, which have not migrated, surrounding the now bright dislocation free cell interiors. This constitutes a contrast reversal of the cell walls and the cell interiors but not substantially the dislocations themselves. Further in Figure 6c, the dislocations now appear to be associated with bright clustering which completes the contrast reversal. Direct comparison of the same region is not possible since the samples are etched and polished during processing.

J. F. Bresse: Why do you think that there are more dark and bright spots in gettered samples?
Authors: In the model that we accept, damage to the back surface introduces new gettering sites which are not saturated with point defects. Point defects can migrate along the dislocations to these new sites. This allows additional point defects to migrate from the bulk to the dislocations increasing the apparent prevalence of the dark and bright spots. Those dislocations that were not previously as effective in gettering now appear.

B. G. Yacobi: In the Introduction you report that "dislocations generally appear as dark spots about 5μm in diameter", and that "...around these dark spots there are very bright rings about 20μm in diameter". In light of the microscopic character of the dislocation, please explain the significance of these numbers (i.e., 5μm dark spots and 20μm bright rings). do these numbers vary? What do they depend on? Are they related to the electronic properties of the material?
Authors: We have not made any correlation of the electronic properties with the observed contrast since no attempt was made to experimentally determine the electronic properties (mobility, lifetime, diffusion length) of this material. The effect of a dislocation core should be on the order of a minority carrier diffusion length giving a limit to the dimension of the central dark core. The numbers quoted are those observed by other investigators. As is clear from the data presented here, the 'dot-halo' patterns do vary significantly (See Figure 8).

B. G. Yacobi: Besides the expected substitutional copper defect (Cu_{Ga}), would you also expect a formation of defect complexes of Cu?
Authors: Other defect complexes are reported in the literature. The Cu_{Ga} defect is the only one of interest since we can observe its luminescence signature.

B. G. Yacobi: Since in some cases the acquisition time of CL images was over 1 hour, did you observe any effect of the prolonged electron irradiation on the CL intensity?
Authors: The overall CL signal is observed to decay due to slight carbon contamination of the surface. The underlying local contrast is not observed to change.

B. G. Yacobi: From the observations of CL images shown in Figure 10 you conclude that the monochromatic CL images corresponding to the band edge excitonic peak (Figure 10a), the carbon-related peak (Figure 10b), and the Cu-related peak (Figure 10c) are all well correlated. However, although there is indeed a general correspondence between them, the lower portion of Figure 10c corresponding to the Cu-related monochromatic CL image is quite different compared to Figures 10a and 10b, indicating possibly somewhat different distribution of Cu.
Authors: Additional background features emerge as the contrast decays in Figure 10 from frame a to c. The additional signal in the lower right portion of the frame is a consequence of the low signal strength and low contrast.