

**PHOTOLUMINESCENCE STUDY OF INTERFACE DEFECTS  
IN BONDED SILICON WAFERS**

W.A. Nevin and D.L. Gay  
BCO Technologies (NI) Ltd., 5 Hannahstown Hill, Belfast, BT17 0LT, N. Ireland

S. Blackstone  
BCO America, P.O. Box 938, Durham NH 03824-0938, USA

V. Higgs  
Bio-Rad Semiconductor Division, Bio-Rad House, Maylands Av.,  
Hemel Hempstead, HP2 7TD, England

**ABSTRACT**

A new photoluminescence technique has been applied to non-destructively image defects at the bonded interface in thick silicon-on-insulator (SOI) and silicon-silicon (Si-Si) bonded wafers. In SOI, the detection and mapping of microvoids was possible with high resolution, confirmed by optical and scanning electron microscopy. In Si-Si, the method was used to compare the interface quality for three different hydrophilic and hydrophobic pre-bond cleans. Transmission electron microscopy and energy dispersive X-ray spectroscopy were used to identify the nature of the observed defects. The results were correlated with SIMS and spreading resistance measurements across the interface, showing that the electrical properties and generated defect type are related to the chemical composition of the interface at join.

**INTRODUCTION**

Understanding the nature of the interface in silicon fusion bonded wafers is important for optimising bonding yields, reducing structural and electronic defects, and improving the electrical performance. For silicon-on-insulator structures, the two main non-destructive analysis methods for observing unbonded regions are optical transmission using infrared illumination and scanning acoustic microscopy (SAM) (1). In the former method, resolution is limited to around 1 mm void diameter, while detection is difficult with highly doped handle materials and very thick buried oxide layers. The SAM technique is capable of higher resolution, but needs long scanning times to achieve this, which is impractical in commercial production, and, since the wafer must be immersed in water, it is difficult to measure as-joined and weakly bonded samples. In addition, neither method is capable of distinguishing the internal characteristics of the voids. We report here a non-destructive technique which enables high-resolution whole-wafer mapping of microvoids in thick-film silicon-on-insulator (SOI) wafers. This method utilises photoluminescence (PL) generated by laser

illumination near the SOI top surface as a probe to create an image of the microvoid via reflection at the interface. Various types of microdefect having different internal structure been identified at the interface, some of which have been characterised using optical and scanning electron microscopy.

We have also used the PL method to study interfacial defects in silicon-on-silicon (Si-Si) bonded wafers. Here, the electrical properties of the Si-Si interface are very important with regard to the performance of structures such as *nn* isotype junctions and *pn* rectifying junctions, required, for example, for integrated gate bipolar transistors (2). The properties have been shown to be influenced by interfacial defects, charges, interfacial oxide, oxide precipitates and contaminants, which are in turn influenced by the pre-join clean, joining atmosphere and post-join annealing conditions (2,3,4). In this work, we have prepared Si-Si samples using hydrophilic and hydrophobic cleans, studied the interface by PL, then further examined the nature of some of the identified defects using transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDX). Chemical analysis of the interfaces using secondary ion mass spectroscopy (SIMS) is also presented, as well as electrical data obtained by spreading resistance (SR) measurements, and the results correlated with the defect analyses.

## EXPERIMENTAL

Silicon-on-insulator samples were prepared by joining polished 5" <100>, 5 ohm-cm, CZ, n-type silicon wafers, followed by a 1050 °C bond-anneal. The buried oxide was a 0.5 µm thermal oxide grown on the handle wafer. The top silicon wafer was then mechanically thinned and polished to around 20-100 µm from the bonded interface. For silicon-on-silicon samples, polished 4" (111), 3000 ohm-cm, FZ, n-type silicon wafers were joined to polished <111>, 0.01 ohm-cm, FZ, n-type handles, using three different pre-join cleans. These consisted of a modified SC-1 last clean (A), a modified SC-1 followed by a modified SC-2 clean (B), each of which left a hydrophilic wafer surface, and a modified SC-1 followed by an HF dip (C), which gave a hydrophobic surface. After bond-annealing at 1150 °C, the samples were further annealed at 1100 °C for 12 h in nitrogen. The high-resistivity layer was then ground and polished to 40 µm thickness.

Photoluminescence measurements were performed using a Bio-Rad SiPHER instrument (5) (Figure 1). This is a non-destructive technique based on photoluminescence, using an optical configuration designed to enhance carrier recombination. A microscopic objective is used to focus light from a laser onto the polished surface of the wafer, the sample is scanned in the X-Y direction, and the emitted luminescence at 1100 nm is collected by the same lens and measured using a sensitive detection system. Defects are normally detected due to the local change in carrier lifetime, and are observed as darkened regions at the physical position of the defect, to give a high-resolution image, which is enhanced by digital filtering. However, as shown below, the detection of interface voids appears to occur via interaction of the voids with light emitted by the silicon material under illumination with the laser probe. The system has the capability to perform whole-wafer maps or high-resolution (1 µm) scans. A CCD camera and white light source were also incorporated to allow imaging of the wafer surface and enable separation of surface and bulk features. Two laser sources were used, operating at wavelengths of 532 nm and 827 nm. Measurements were made at room temperature in air.

The Si-Si bonded interfaces were electrically characterised by spreading resistance measurements (Semiconductor Assessment Services) across a bevelled 60  $\mu\text{m}$  thickness. To examine contaminants at the interface, SIMS analysis (MATS UK) was performed for C, O, F, B, S, Na, Mg, Al, K, Ca, Cu and Fe, on samples in which the high-resistivity top Si layer was thinned to 4  $\mu\text{m}$ . Transmission electron microscopy in combination with energy dispersive X-ray spectroscopy (Marconi Materials Technology) were used to identify the nature of the interfacial defects observed by photoluminescence scanning.

## RESULTS AND DISCUSSION

Whole-wafer PL scanning was carried out on SOI using a probe laser wavelength of 532 nm. This detected microvoids of less than 0.5 mm diameter as dark or bright circles superimposed on the background PL signal. High-resolution scanning of these defects showed them to be of several types, but predominantly of two kinds: completely dark (non-luminescing) circles, and dark circles containing brightly luminescing circles at the centre. Similar results were obtained with all SOI thicknesses examined (20-100  $\mu\text{m}$ ), although it was found that with the thickest layers, the sensitivity was improved by switching to the 827 nm laser probe. Figure 2 shows a typical example of a 300  $\mu\text{m}$  diameter void of the second type, taken using the 532 nm laser probe, in a 20  $\mu\text{m}$  SOI. To confirm that these were voids at the bonded interface, samples were thinned to less than 2  $\mu\text{m}$ , the surfaces tape-pulled to remove unbonded silicon, and the positions of the defects compared with those recorded on the PL scan. By using the built-in optical microscopic imaging system in conjunction with scanning electron microscopy (SEM), it was possible to directly compare the PL image and optical image for the same void. For example, the void in Fig. 2 was found to have a fused remnant of the top silicon layer at its centre, indicating that it had been caused by a particle or spike on one of the wafers. The unbonded area is characterised by a weak PL signal, and the central joined area is strongly luminescent. Thus, while further work is needed to clarify the structures of the various other microdefects imaged by the PL, it is apparent that the method can be used to give information on the internal structure of the voids, aiding identification of their origin in a manufacturing environment.

Since the depth detection limit for the SiPHER technique has been demonstrated to be approximately 1  $\mu\text{m}$  (5), it is surprising that features at the interface, 20-100  $\mu\text{m}$  below the surface, can be clearly imaged. It is thought that the image results from penetration of the photoluminescent light, generated near the surface of the SOI layer, to the interface. This is then reflected back at the bonded interface, or it is scattered at an unbonded void, to give a reduction in detected PL intensity. The technique is therefore applicable to routine detection of sub-millimetre microvoids in SOI material.

Figure 3 compares high-resolution PL images of Si-Si samples prepared using the three different pre-join final cleans, taken with a 827 nm laser probe (it was found that for these samples, the 827 nm probe gave better resolution than 532 nm). Three different features are clearly resolved at or near the interface. The highest density of defects is observed for the SC-1 last clean, consisting of bright circular islands with about 100  $\mu\text{m}$  diameter, dark wavy lines and dark, straight lines. The SC-2 last clean contains a similar density of the circular and straight-line features, but much lower density of wavy defects. In contrast, the HF-last clean shows a marked reduction in all three types of defect.

SR measurements across the interface for these samples showed increasing deterioration of the electrical properties of the interface on going from the HF-last clean to the SC-1 clean. The results for the HF and SC-1 cleans are shown in Figure 4. HF-last gives an excellent electrical interface, while a p-type inverted layer is identified at the interface for the SC-1 clean. The results for the SC-2 clean were intermediate, showing an intrinsic region instead of a p-type peak in the high-resistivity layer next to the interface. SIMS analysis indicated metallic contamination of mainly Al, Ca, Cu and Na for the sample with the SC-1 last clean. As expected, the metal contamination was reduced to low levels for the SC-2 and HF cleans, while a small amount of fluorine was detected at the interface with the HF-last clean. From these results it is likely that the poor interfacial electrical characteristics of the SC-1 clean are related to the diffusion of contaminants from the interface during the high-temperature anneal.

TEM was used to examine the defects in the sample with the SC-1 clean. This showed that a 2 nm thick interfacial oxide present after bonding had broken up into 6 nm thick clustered regions, which correspond to the bright circular features of the PL image. This is in agreement with the observation that these are seen for the two hydrophilic cleans, but not in the hydrophobic sample, which should not have an interfacial oxide layer. Previous work on Si-Si hydrophilic bonding has shown that the interfacial oxide starts to break up into islands at temperatures of around 1100 °C (6). At positions where the oxide has been broken up, screw dislocations are observed at the interface, as shown in Figure 5. These are generated to relieve the stress of the lattice mismatch due to misalignment of the wafers, as has been seen previously in hydrophobic Si-Si bonding (3). Figure 6 shows a TEM image of the wavy-type feature seen by PL. These are dislocations extending into the bulk of the top Si layer, which have straight segments and shorter curved segments where the dislocation deviates from the straight line. Many small precipitates can be seen in the region between the curved segment and the line joining the straight segments. This is typical of dislocation climb in conditions of impurity contamination. The dislocations are initially introduced by slip and lie along the (110) directions. Impurity atoms then precipitate on the dislocation during annealing, pinning the dislocation along its length. Regions of dislocation that break free from the precipitates can climb out of the straight-line configuration, and precipitation in the new position then occurs. The origin of these dislocations is not clear. They may originate from the interfacial defects at the oxide islands, or possibly by gliding from the edges of the wafer. The chemical composition of the precipitates was examined by EDX (Figure 7), and found to contain mainly copper. The copper has probably originated from the interfacial contamination, identified by SIMS, since copper can diffuse rapidly through silicon at the annealing temperatures used. This is consistent with the decrease in density of the wavy dislocations in samples with the SC-2 or HF last cleans, which had low metallic contamination at the interface.

## CONCLUSIONS

Photoluminescence has been shown to be a useful non-destructive technique for studying defects at the bonded interface in thick SOI and Si-Si structures. In the case of SOI, a whole-wafer map could be obtained with sub-millimeter resolution of microvoids. High-resolution scanning of areas of interest was then used to distinguish between several different types of void, and also gave information on the internal structure of these voids.

This method therefore appears useful for routine detection of microvoids in SOI in a production environment.

In addition, we have used the technique to investigate the interface quality for Si-Si wafers bonded using three different pre-bond cleans, in conjunction with TEM, EDX, SIMS and SR measurements. For both SC-1 and SC-2 hydrophilic bonding, the PL analysis identified circular oxide agglomerates, not present with HF-last hydrophobic bonding, which have resulted from the break-up of the interfacial oxide during high-temperature annealing. Associated with these were screw dislocations and slip, generated by stress at the resulting oxide gaps. For the SC-1 last clean, the PL measurements also showed a high density of threading dislocations with associated copper precipitates, which were not present for either the SC-2 or HF clean. In agreement, the SIMS analysis confirmed much lower levels of metallic contamination at the interface, as expected for these cleans. The SR data showed an improvement in the electrical properties of the interface on going from SC-1 to SC-2 to HF, in correlation with the decrease in interfacial defect density observed by PL.

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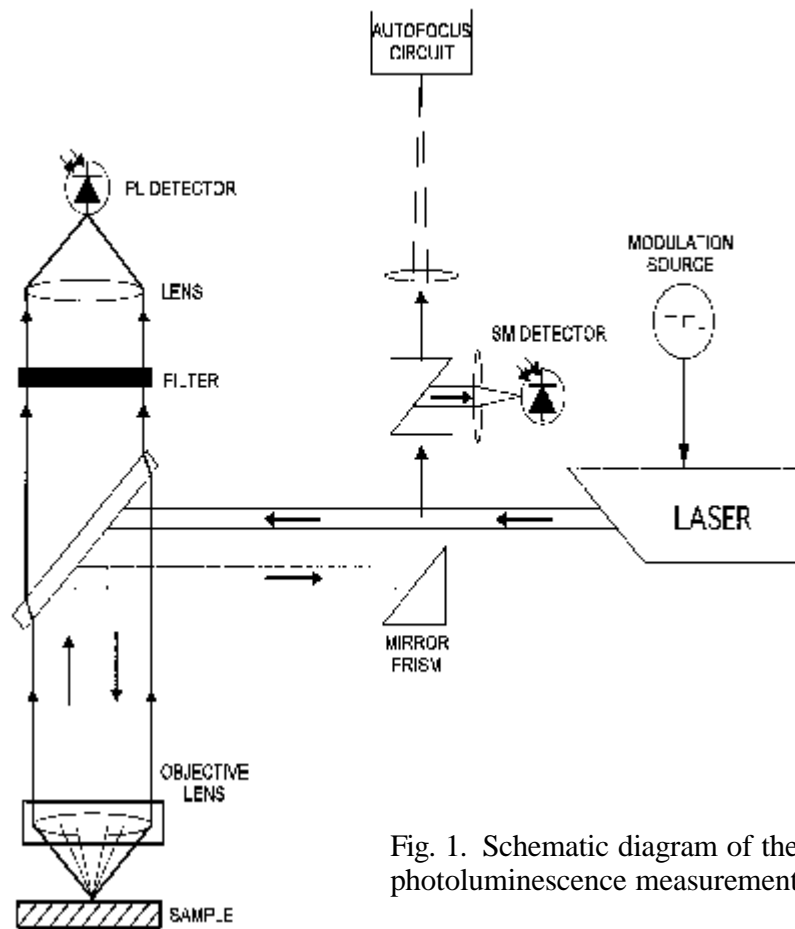


Fig. 1. Schematic diagram of the photoluminescence measurement system.

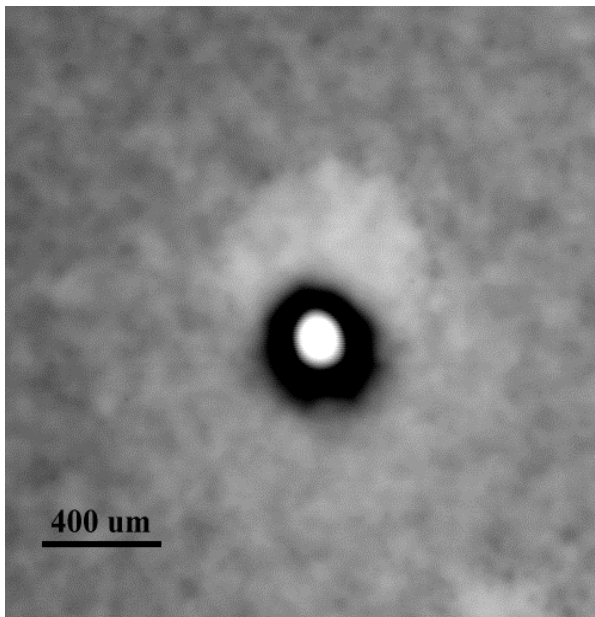


Fig. 2. Photoluminescence image of a microvoid at the interface of a 20 μm thick SOI layer, taken using a 532 nm laser probe.

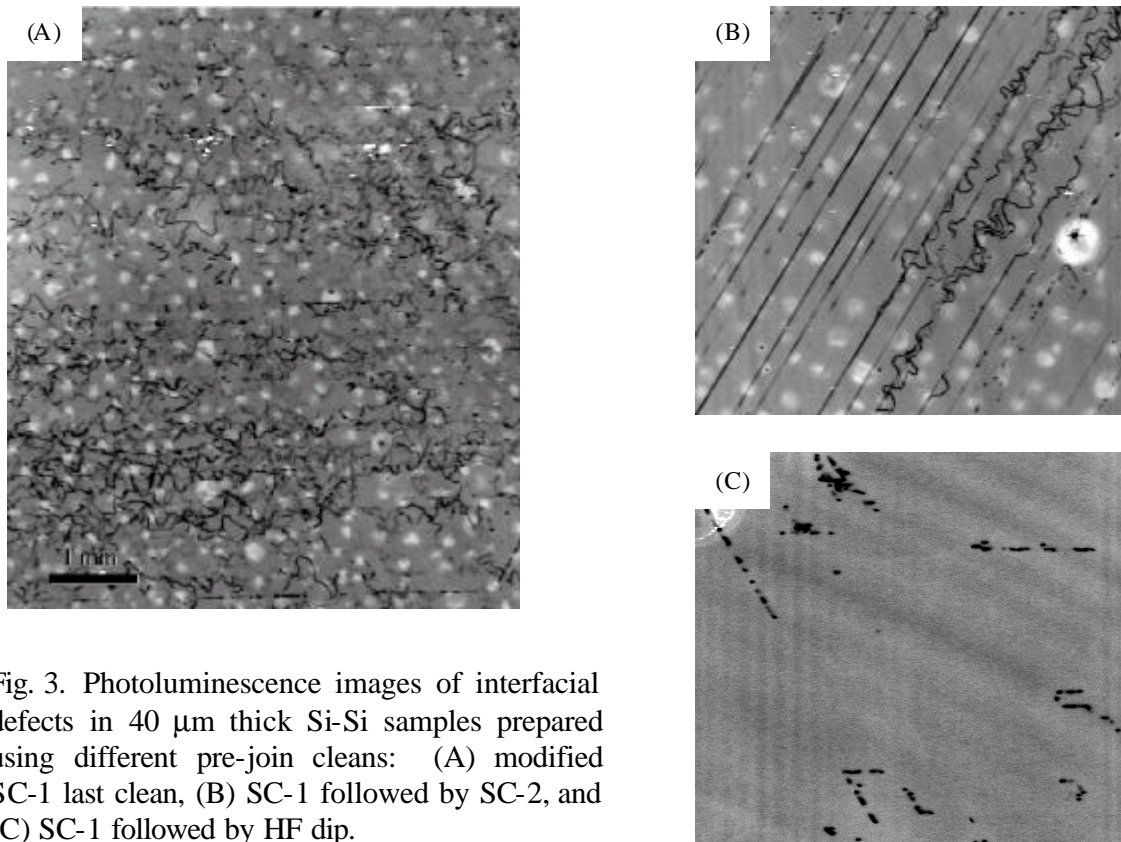


Fig. 3. Photoluminescence images of interfacial defects in 40  $\mu\text{m}$  thick Si-Si samples prepared using different pre-join cleans: (A) modified SC-1 last clean, (B) SC-1 followed by SC-2, and (C) SC-1 followed by HF dip.

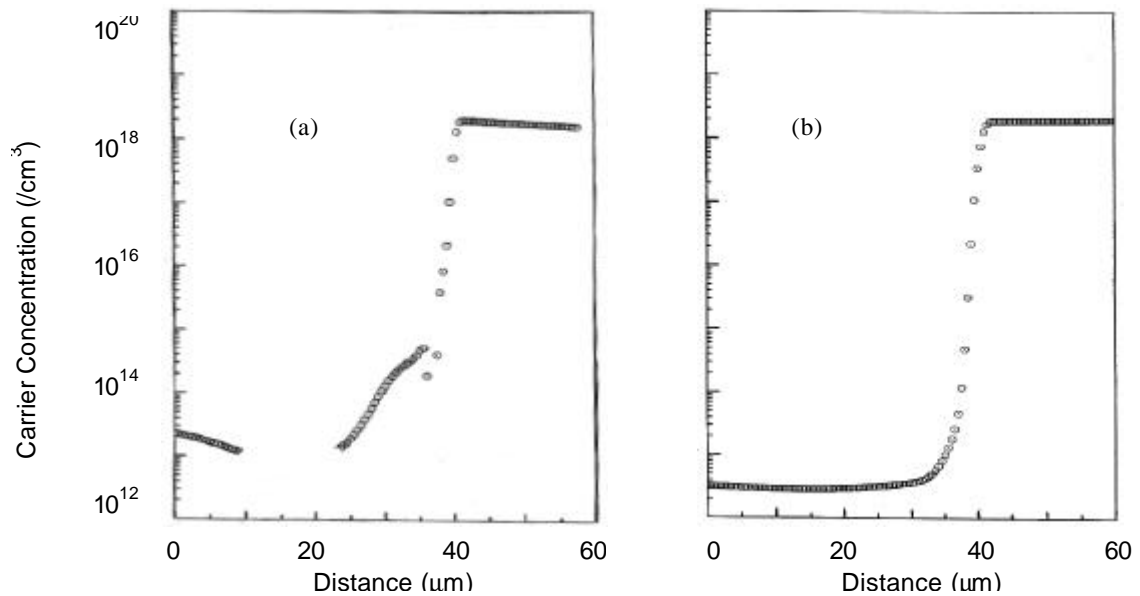


Fig. 4. Spreading resistance profiles for Si-Si samples prepared using (a) SC-1 last pre-join clean, and (b) SC-1 followed by HF dip.

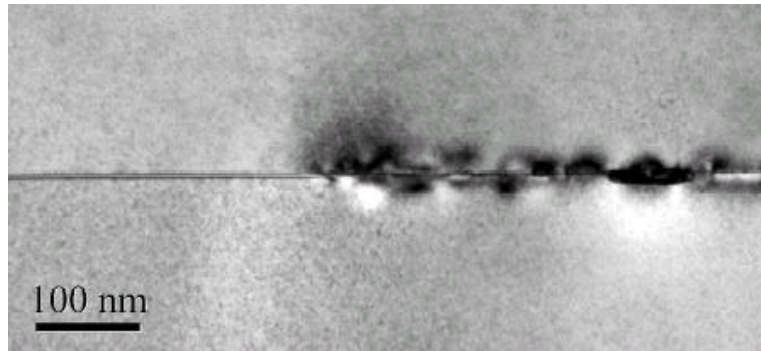


Fig. 5. Cross-sectional TEM image of an oxide island and associated screw dislocation at the interface of a hydrophilically bonded Si-Si wafer.

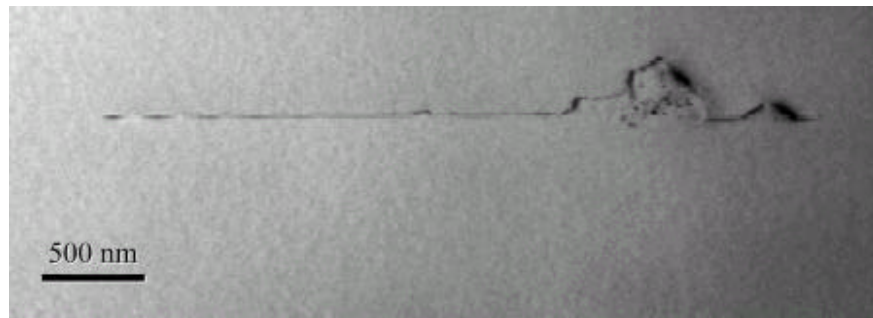


Fig. 6. TEM image of a threading dislocation with associated precipitates in a Si-Si sample prepared using an SC-1 pre-join clean.

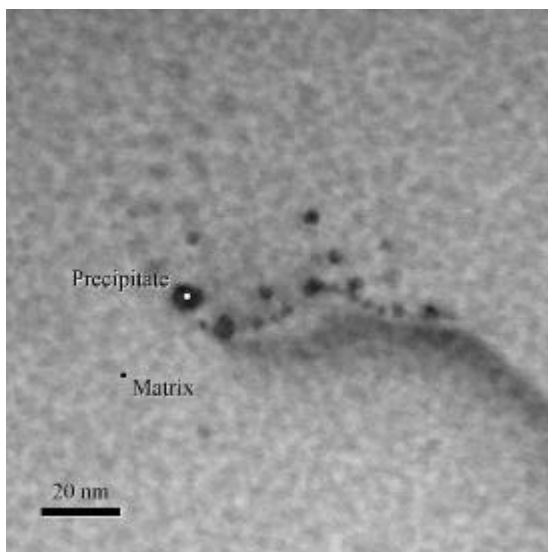


Fig. 7. Bright field, cross sectional TEM image of Cu precipitates on a threading dislocation in the sample of Fig.6. Regions of analysis are marked by circles with the same diameter as the electron probe size.