FIB preparation and TEM characterization of Si_3N_4 precipitates grown in multicrystalline Si for solar cell application

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During processing of block-cast multicrystalline silicon (mc-Si), Si_3N_4 and SiC precipitates appear frequently at the top of mc-Si blocks [1,2]. There are three different types of Si_3N_4 precipitates: 1) hexagonal-shaped Si_3N_4 rods surrounded by SiC particles (Figure 1(a)), 2) Si_3N_4 filaments growing at grain boundaries of mc-Si (Figure 1(b)) and 3) Si_3N_4 filaments growing outside of grain boundaries of mc-Si (Figure 1(c)). The first and second types of these precipitates are already well characterized by scanning electron microscopy (SEM) equipped with an energy dispersive X-ray spectrometer [1,2] and by electron backscattered diffraction [1]. However, due to the complexity of specimen preparation, the reports on characterization of the precipitates by transmission electron microscopy (TEM) are very rare. It is well-known that the different thinning rate in two phase materials, e.g. Si_3N_4 embedded in mc-Si, is a common problem in the samples preparation for TEM investigation by conventional methods. This problem can be overcome by using focused ion-beam (FIB) for preparation.

In the current study, we have used a FIB milling in the samples preparation for TEM investigations as described in Reference [3]. To prepare a cross-sectional specimen a FIB lamella was cut perpendicular to the area of interest (Figure 2(a)). To produce a plan-view sample a FIB lamella was cut parallel to the area of interest (Figure 2(b)). TEM investigations were carried out using a CM 20 Twin (Philips, Netherlands) microscope operated at 200 kV.

Figure 3(a) shows a cross-sectional bright field TEM image and a selected area electron diffraction (SAED) pattern (inset), respectively, of a Si_3N_4 rod. The patter is indexed according to the β modification of Si_3N_4. The TEM investigations also show that the rod is single crystalline and is growing in [0001] direction of Si_3N_4. Figure 3(b) shows a cross-sectional bright field TEM image and a SAED pattern (inset), respectively, of a Si_3N_4 filament growing at a grain boundary of mc-Si. The structural modification of the filament is found to be the α modification of Si_3N_4. The TEM investigations also show that the filament is single crystalline and contains defects like grain boundaries. Figure 3(c) shows a plan-view bright field TEM image and an SAED pattern (inset), respectively, of a Si_3N_4 filament precipitated outside of a grain boundary of mc-Si. The patter is indexed according to the β modification of Si_3N_4. No accumulation of dislocations is found in the Si matrix around the Si_3N_4 filaments.

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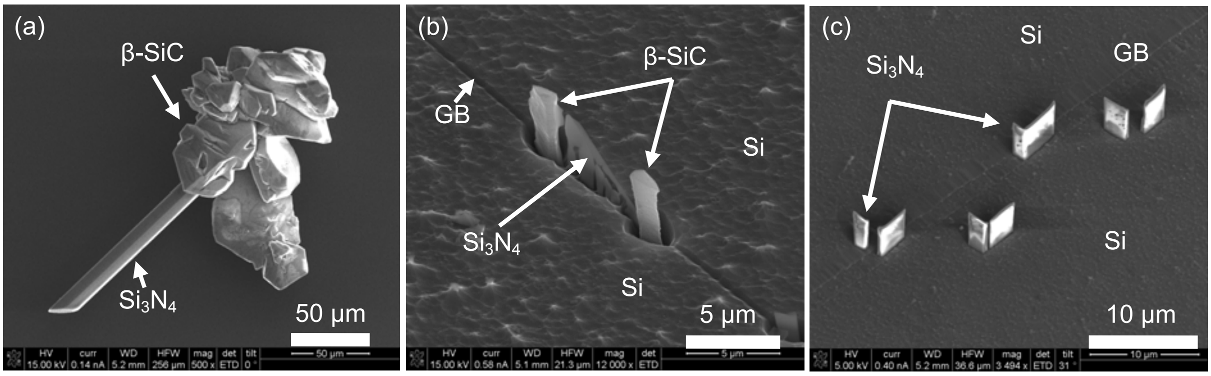

Figure 1. (a) SEM image of a Si_3N_4 rod surrounded by a cluster of β-SiC particles, (b) SEM image of Si_3N_4 and β-SiC filaments growing at a grain boundary (GB) of mc-Si and (c) SEM image of Si_3N_4 filaments growing outside of grain boundaries of mc-Si.

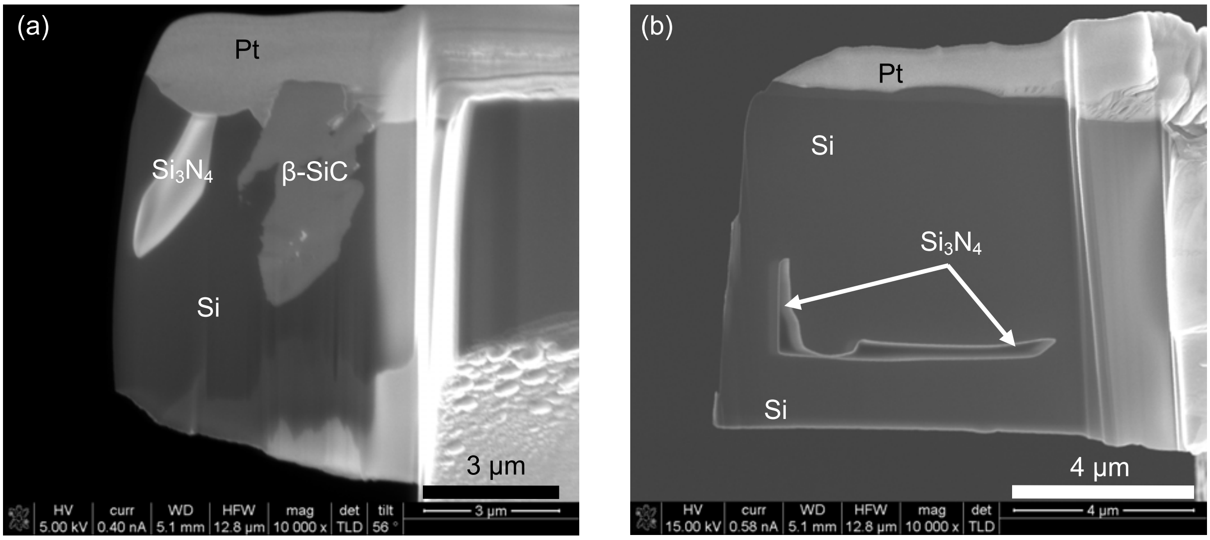

Figure 2. (a) SEM image of a FIB lamella (cross-sectional method of preparation) prepared from a sample containing Si_3N_4 and SiC filaments precipitated at grain boundaries of mc-Si like in Fig. 1(b) and (b) SEM image of a FIB lamella (plan-view method of preparation) prepared from a sample containing Si_3N_4 filaments precipitated outside of grain boundaries of mc-Si (see Fig. 1(c)).

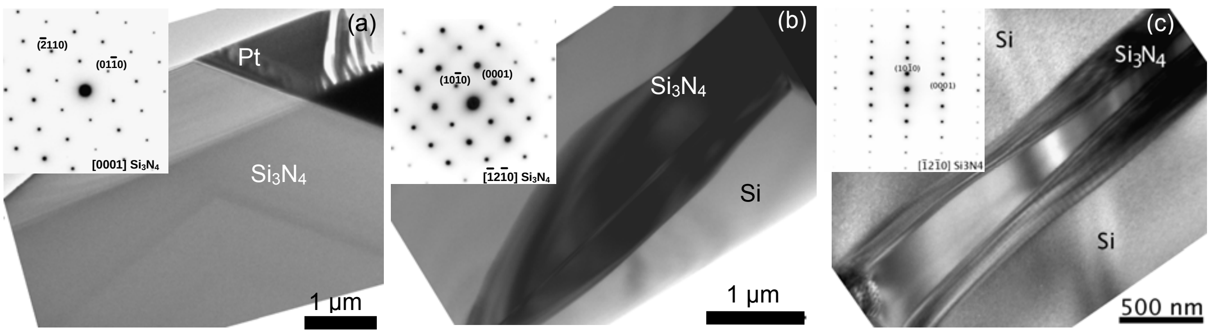

Figure 3. (a) Cross-sectional bright field TEM image of a Si_3N_4 rod. The inset shows the SAED pattern of the rod, (b) cross-sectional bright field TEM image of a Si_3N_4 filament growing at a grain boundary of mc-Si. The inset shows the SAED pattern of the filament and (c) plan-view bright field TEM image of a Si_3N_4 filament precipitated outside of a grain boundary of mc-Si. The inset shows the SAED pattern of the filament.

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