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Layer and Defect Structures of BaF_2/CaF_2 Multilayers

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Nanocrystalline ionic conductors attract increasing interest in recent years.[1,2] Multilayers of BaF_2/CaF_2, e.g., show significant dependence of the conductivity (effective conductivity parallel to the interface) on the layer thickness, *l*.[3] When *l* is larger than 7.5nm, the conductivity increases progressively with increasing interface density, especially an anomalous increase of conductivity is found in the range of $7.5< l <50$nm. When *l* is smaller than 7.5nm the conductivity decreases. The questions thus arise, how the scale of heterolayers may influence their structures and the generation of defects, and how these relate to the conductivity.

BaF_2 and CaF_2 multilayers with various thickness, *l*, have been grown by MBE on $(01\bar{1}2)$ sapphire.[3] Special care has been taken in preparing cross-sectional TEM specimens due to easy damage by Ar^+-ions during ion-milling and weak adhesion between the multilayers and the substrate. The image processing package LADIA [4,5] was used for measuring the lattice distances.

Fig. 1 shows the layer structures of samples with $l = 40, 10, 1$nm. The BaF_2 and CaF_2 layers are well distinguished by dark and bright contrast. Electron diffraction reveals the orientation relationship between the layers and the substrate: BaF_2/CaF_2 (111)||Al_2O_3 $(01\bar{1}2)$, and BaF_2/CaF_2 $[11\bar{2}]$||Al_2O_3 $[2\bar{1}\bar{1}0]$. The interfaces between layers are wavy, especially when *l* is smaller, which explains moiré fringes often observed close to the interfaces. In the case of $l = 1$nm, column-like structure forms. Within an individual column layers are well deposited, as proved by the HAADF image in Fig.2. The continuity of the layers is distorted at column boundaries, which may impede ion transport parallel to the layers. Dislocations are observed either by diffraction contrast, as arrowed in Fig.1(b), or by HREM (Fig.3 and Fig.4), where the extra atomic half-planes are marked with $\perp$. Fig.3 illustrates a row of $[11\bar{2}]$ dislocations with a Burgers vector of $a/2\ [\bar{1}10]$, forming a low-angle boundary along [111]. In the case of $l = 10$nm, interface dislocations are always present in the CaF_2 layers (Fig.4) with Burgers vectors parallel to [111], demonstrating a novel relaxation mechanism in this lattice-mismatched heterostructure. In order to investigate whether intermixing takes place between the layers, (111) lattice spacings are measured (Fig.4). The abrupt changes of the lattice spacings between adjacent layers indicate that the intermixing between wavy BaF_2 layers and CaF_2 layers may be negligible. Interesting to note is that the lattice spacings in CaF_2 layers are markedly smaller (~15%) in the vicinity of their interfaces than inside the layers. This is attributed to the interface dislocations described above. In addition to the difference in the chemical nature, lattice mismatch between the layers, local interface structures and dislocations may influence charge redistribution, and thus the conductivity anomaly.

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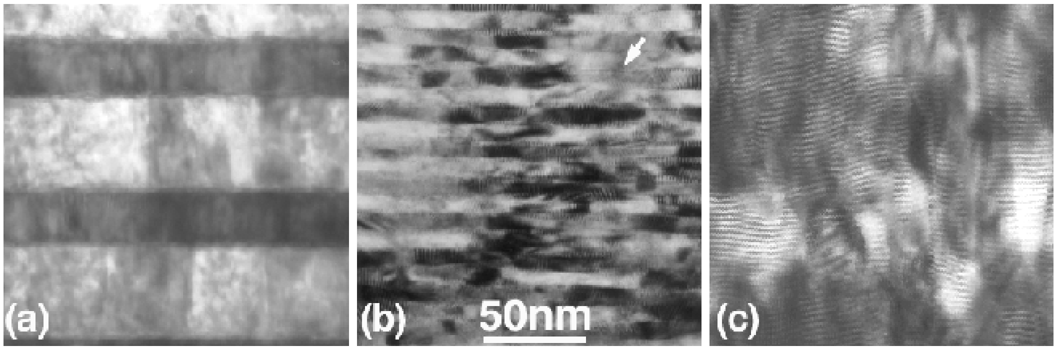

Fig.1 Bright-field image of BaF_2/CaF_2 multilayers, l = 40nm (a), 10nm (b) and 1nm (c).

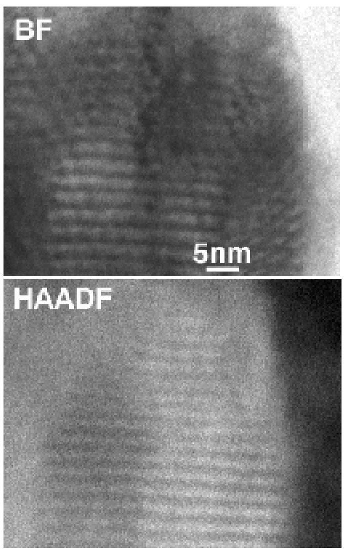

Fig.2 Images from specimen C.

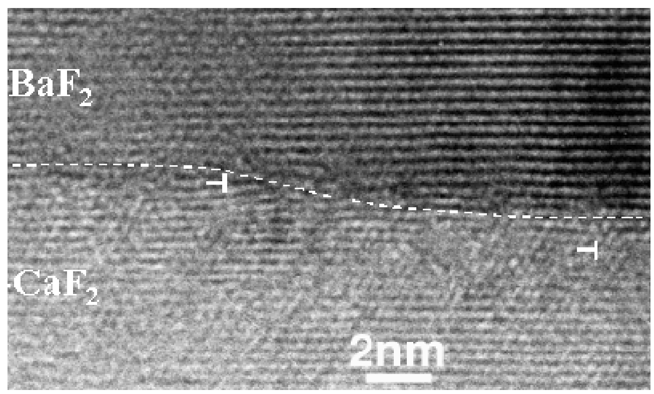

Fig.4 Interface dislocations lying in CaF_2 (specimen B).

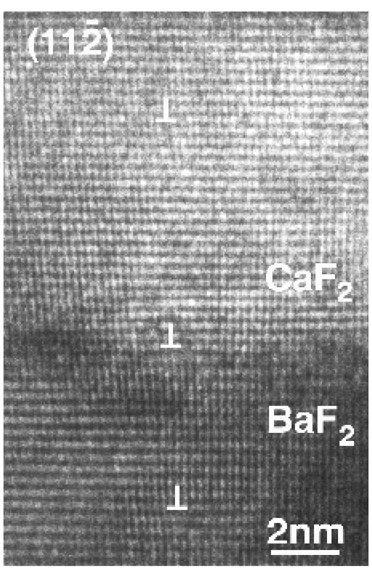

Fig.3 Low-angle boundary in specimen B.

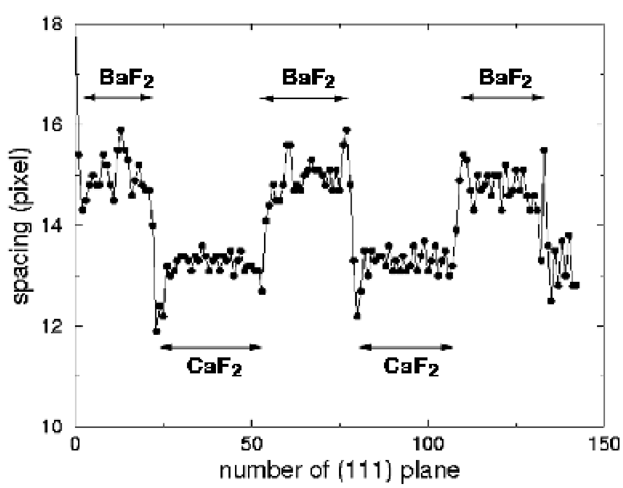

Fig.5 (111) spacings measured from specimen B.

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