

Current Science approached the Directors of the National Science Laboratories in India in December 2014 with a request to briefly highlight the most significant researches carried out in their Laboratory during the year. Starting from this issue, we present a brief summary of the most significant researches carried out in our National Laboratories.

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Realization of single-terminated nano step-and-terrace-like surface of SrTiO₃ single crystals: Institute of Nano Science and Technology, Mohali

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Oxide interfaces and superlattices are of growing interest because of their potential in next generation spin-electronic devices. To obtain high quality oxide interface and superlattice, atomically flat and well defined substrate surface is essential. We have developed a mild and chemical free process through which such high quality step-and-terrace-like surface of perovskite substrates can be achieved.

Keywords: Atomic stacking, clean interfaces, perovskite oxide, single crystals.

OXIDE materials are of key interest because of the fundamental physics related to them and their potential for diverse practical applications. This class of material displays complex phase diagrams and competing ground states owing to the presence of strong electronic interaction and spin-orbit coupling, which makes them sensitive to external parameters, such as magnetic field, strain, light, etc.

Among oxides, perovskite materials (ABO₃) are particularly interesting because of their simple crystal structure and versatile physical properties¹. If two or more perovskite oxides are coupled together to form an interface, then integrated or even new properties emerge which cannot be observed in the bulk²⁻⁷. The realization of such oxide interface is, however, often hampered by the presence of defects in oxides⁸. Yet, developments in the epitaxial oxide growth techniques are capable of realizing such clean interfaces⁹. One of the crucial criteria to achieve extremely clean interface is to use single-terminated, single-crystalline and defect-free substrate, where the surface of the substrates is atomically well

defined. The growth kinetics and overall quality of the materials are significantly dependent on the initial surface quality.

One of the important substrate materials is SrTiO₃ (STO), which is commonly used because of its good chemical compatibility and lattice matching with other

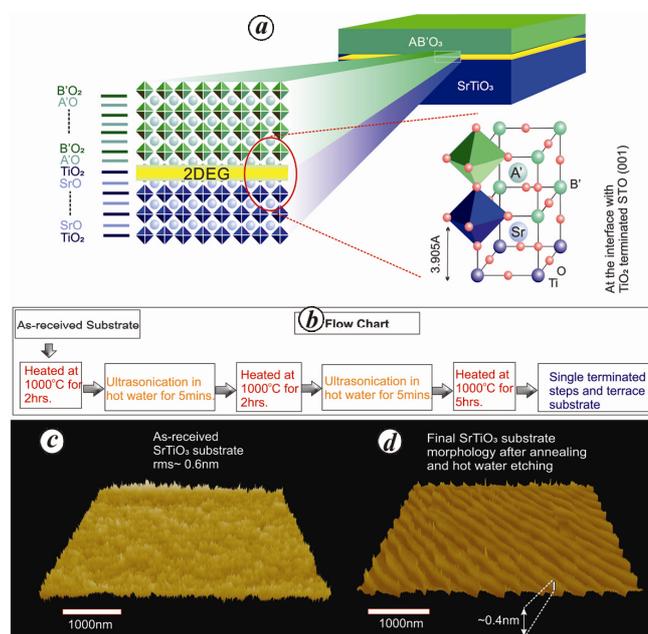


Figure 1. (a) The interface between SrTiO₃ and other perovskite materials ABO₃. Possibility of the formation of novel two-dimensional electron gas (2DEG) at the interface is also shown. The atomic stacking layers have been indicated on the left side. Enlarged view of the crystal structure near the interface is shown on the right side. (b) A schematic flow chart of the annealing and etching used to achieve single-terminated step-and-terrace-like surface. (c) AFM image of the substrate as-received. (d) AFM image of the substrate after all the steps of annealing and etching treatment. Terrace step height is ~0.4 nm.

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materials⁹. The interface between STO and other materials such as LaAlO₃, LaVO₃ and LaTiO₃ has shown diverse emergent phenomena such as the formation of two-dimensional electron gas, quantum oscillation in resistivity, superconductivity, etc.^{5–7}. Figure 1 *a* shows a schematic diagram of the interface between STO (001) and another perovskite ABO₃. An enlarged view of the interface with atomic stacking layers is also presented on the left side of the figure. STO (001) has two atomic stacking layers – TiO₂ and SrO. It has often been observed that novel properties could be realized if the STO (001) has only TiO₂ terminated surface. In Figure 1 *a*, we have assumed that the STO substrate is TiO₂-terminated.

Commercially available STO (001) substrates are usually mixed terminated and till date, researchers are using buffered HF solution to etch SrO from the surface¹⁰. Buffered HF solution is an aggressive chemical, which often leads to uncontrolled etching, introducing holes on the surface which obstruct thin film growth. Hence, a careful treatment is necessary to get reproducible high-quality surface.

We have recently shown a controlled, reproducible and mild etching technique through hot water ultrasonication method, which can produce high-quality single-terminated terraces separated by mono-atomic layers of STO (001) substrate (Bhanu Prakash and S. Chakraverty, unpublished).

Commercially available and one-side mechanically polished substrate has been used as a starting material. Figure 1 *c* shows atomic force microscopy (AFM) image of an as-received substrate. Small corrugation with rms roughness ~0.6 nm has been observed from AFM image with no signature of steps-and-terraces. The substrate was then heated to 1000°C for 2 h to facilitate the surface recrystallization in order to achieve step-like surface structure. After annealing, formation of particle-like agglomeration on the substrate surface was observed, which could not be removed by simple ethanol–acetone cleaning. Whereas those particles disappeared after the substrate was ultrasonicated in hot water. This high-temperature annealing followed by hot-water etching process was repeated three times. The final annealing was done for relatively longer time (5 h). This process is

schematically described in Figure 1 *b*. Figure 1 *d* shows the AFM image of the STO (001) surface after the whole process is completed. No trace of corrugation was observed and clear steps-and-terrace-like structure with terrace height of ~0.4 nm (lattice parameter of STO (001) is 3.905 Å) was achieved.

In conclusion, we have shown a novel way to achieve single-terminated, nano step-and-terrace-like surface morphology of STO (001) substrate through a simple and mild hot water selective etching technique combined with high-temperature annealing process. This technique may pave the way to achieve single-terminated surface of the materials containing reactive elements. We are further exploring the possibilities of preparing atomically well-defined surface of such perovskite oxides.

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ACKNOWLEDGEMENTS. We thank Ashok K. Ganguli, Director, Institute of Nano Science and Technology, Mohali for encouragement, support and fruitful discussions during this work. The support provided by DST, New Delhi is also acknowledged.