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Summary

Currently, scanning probe microscopy and nanoindentation are two widely used techniques for the characterization of materials at nanoscale. These techniques are used to investigate physical, chemical, thermal, electrical and optical properties of a material. Although these techniques have contributed significantly to material research, their use outside the research laboratory is restricted due to the complexity of the instrument. Hence, novel developments that bring about step change in instrumentation and functionality of these techniques can help to expand their application areas.

The work presented in this thesis focuses on the development of a new technology platform – *fiber-top* technology, as a tool for material property characterization at nanoscale. The research done towards the development of this technology was highly multidisciplinary and encompasses areas of optic fiber sensing, micro-technology/micro-fabrication, control systems, optics and biomaterials. The thesis work can broadly be divided into two important application areas of *fiber-top* technology: (i) Scanning probe microscopy and (ii) Nanoindentation.

The concept of *fiber-top* technology – carving a cantilever on top (cleaved end) of an optical fiber was introduced by D. Iannuzzi *et al* in 2006. In such a *fiber-top* device, a laser when shone from the other end of the optical fiber, travels along the optic fiber core and emerges at the face of the optical fiber. Part of this light reflects back and a part transmits further to be reflected by the surfaces of the cantilever. These reflected signals travel back though the same optical fiber and are redirected on a photodiode. With this detection scheme, the deflection of the *fiber-top* cantilever can be measured with sub-nanometer resolution.

In the first chapter of this thesis, the background and the principle of scanning probe microscopy are discussed, followed by an introduction to the concept of *fiber-top* technology, its detection scheme, working principle and limitations. The next chapter introduces a novel cost effective fabrication process as an alternative to *fiber-top* technology. In this new concept called *ferrule-top* technology, a cantilever is fabricated on top of a ferruled optical fiber. The chapter also discusses the fabrication of a ferrule-top atomic force microscope (AFM) probe and the construction of a fully functional AFM setup. This AFM setup successfully demonstrates contact mode imaging of a sample in air and liquid environment.

The third chapter incorporates improvements in the ferrule-top probe fabrication process and further extends its capabilities as a versatile tool for nanoscale imaging. Here, in the first part of the chapter we demonstrate tapping mode and contact mode imaging performed at 12 degree Kelvin. In the second part, we integrate the ferrule-top probe into a high speed AFM scanner and perform contact mode imaging up to 2 frames/second. The fourth chapter focuses on an important and unique capability of *fiber-top* technology of combining scanning probe microscopy with near field optical microscopy (SNOM), i.e. able to simultaneously investigate topography and optical property of a sample. Here we demonstrate two different approaches adopted for the fabrication of a *fiber-top* and a ferrule-top probe with near field capabilities. The results obtained demonstrate that the *fiber-top* and the ferrule-top technology can provide multiple advantages over the conventional SNOM techniques.

The work presented in the fifth chapter focuses on the development of ferrule-top technology for application area of Nanoindentation. Ferrule-top technology provides unique advantages over conventional indentation instruments in terms of better force resolution, easy of handling and ability to perform indentation testing in liquid environment. The results obtained from indentation testing of various polymers demonstrate the full functionality of a ferrule-top indentation probe. In the following chapter, we extend the capability of ferrule-top probe to perform optical coherence elastography (OCE) by combining indentation and optical coherence tomography (OCT) in a specially fabricated ferrule-top probe called OMNE (OptoMechanical NanoElastography) probe. This probe allows one to impart calibrated load with nanonewton force resolution to a sample and simultaneously observe the material deformation within the sample during the indentation stroke.

The final chapter presents some of the latest developments of ferrule-top nanoindenter to accommodate large sample sizes and scan areas, as a step towards preclinical indentation trials. Furthermore, this chapter also discusses other areas of applications wherein ferrule-top technology can be used to study fundamental biological processes by means of mechanotransduction and chemiluminescence. Finally, *fiber-top* technology that was invented serendipitously has matured tremendously over the years and is being commercialized by a spin-off company (Optics11 BV).