9.1 Introduction

Since tin whiskers were discovered by Compton et al.,
whiskers have attracted special attention of many investigators for their characteristic forms and physical properties such as mechanical strength. Advances in scientific, industrial and military products are increasingly dependent on the availability of new and improved structural materials that have high specific strength, modulus and high temperature resistance. The whole impetus behind current research on whisker composites lies in the fact that whiskers are strongest form of solid yet discovered. Also,
the reason for adopting whisker crystals is that they have a better degree of perfection compared to the bulk crystals. Several methods have been used to grow alkali halide whiskers e.g. growth within solution, cleavage of bulk crystals, growth in oven, growth from porous substrate and growth from porous membranes. Growth of magnesia whiskers and metal whiskers have been reported by vapour phase method.

The present chapter deals with growth, characterization, morphology and possible mechanism of growth of SnI₂ whisker crystals by gel technique.

9.2 Experimental and Observations

9.2.1 Whisker growth

Experimental system and procedure for preparation of gel are same as described in chapter 5. However, data relating to the whisker growth of SnI₂ is as under:

Sp. Gr. of sodium meta silicate solution = 1.04
Concentration of impregnent solution in gel, SnCl₂ = 1.0 N
Concentration of supernatant solution, KI = 2.0 N
pH of the gel mixture before setting = 3.
growth of whisker clusters growing parallel to the b-axis, and a process of space filling between the whiskers. Such an array of whiskers are also observed in CdS\textsuperscript{14}) and ZnS\textsuperscript{15}). The opening in the prism faces are explained through a possible difference in the velocity of growth of whiskers\textsuperscript{6}).

The morphological study of whiskers make it possible to distinguish the growth of whiskers by stages, as in growth of inorganic salts in supersaturated water solution\textsuperscript{17}). The first stage can be characterized by a rapid linear growth and during second stage broadening of whisker takes place through layer growth surrounding the faces. The separate layers originate usually at the base and grow in the direction of the tip, forming a fine striation on the walls of whisker. The observed striated structure can be related with the thickening stage of whisker growth.

The few traces of calcium, potassium and chlorine have been identified as impurities in SnI\textsubscript{2} and SnI\textsubscript{4} crystals during their electron microprobe analysis. This strongly suggests that above impurities in the solution are supposed to be of importance for the growth of hollow whiskers\textsuperscript{16}).
9.5 **Variation of Fracture Strength**

When a whisker having thickness \((t)\) and Young's modulus \((E)\) is bent into a loop of radius \((r)\), then the strain \((\epsilon)\) at the surface is given by\(^{18}\)

\[
\epsilon = \frac{t}{2r}
\]

and the stress \((\sigma)\) at the surface is

\[
\sigma = \epsilon E = \frac{Et}{2r}
\]

The radius \((r)\) is reduced until fracture occurs, \(\sigma\) then represents the ultimate tensile strength and \(\epsilon\) the ultimate extension.

The uniform whiskers were fixed firmly to polyethylene sheets with quick fix and then were uniformly bent perpendicular to the whisker axis. The whiskers were kept under observation in an optical microscope and were gradually bent until they fracture. The radius of curvature and the whisker thickness at the point of fracture were measured with the help of the fiber-eye piece attached to an optical microscope. The plot of the radius of curvature \((R)\) on fracture against the whisker thickness \((t)\) is shown in fig. 9.16. The plot is a straight line with uniform slope in the range of 30 to 220 micron thickness of the whiskers. Since the slope is constant, the fracture strength \((\sigma = Et/2r)\) is constant for the whiskers and no variation of it with thickness is exhibited unlike in
the case of silicon carbide and tungsten crystals\cite{19,20} for which the strength rapidly increases with a decrease in thickness for low values of whisker thickness.

9.6 Conclusions

1. The solid and hollow whisker crystals of SnI\(_2\) as long as 10 mm have been grown by gel technique.

2. Needle shaped and ribbon shaped whiskers have been observed throughout the growth.

3. SnI\(_2\) whiskers had \(\langle 010\rangle\) orientation and were bounded by \(\{010\}\) faces.

4. The observations suggest that hollow whiskers formation is possible with or without the presence of impurities.

5. By using electron microprobe analysis, the overgrown micro-crystallites have been identified as SnI\(_4\) crystals on the SnI\(_2\) whisker crystals.

6. Two dimensional nucleation appears to be the responsible mechanism for the growth of SnI\(_2\) whiskers.
7. The straited structure on the surface of whiskers can be related with the thickening stages of whisker growth.

8. The ultimate tensile strength of SnI$_2$ whiskers is independent of its thickness.
References


Captions of the figures

Fig. 9.1 Growth of SnI\(_2\) whiskers inside the silica gel.

Fig. 9.2 Growth rate of SnI\(_2\) whiskers.

Fig. 9.3 Growth of SnI\(_2\) whiskers along with SnI\(_4\) crystals inside the silica gel.

Fig. 9.4 (a) Needle shaped SnI\(_2\) whiskers.

Fig. 9.4(b) Ribbon shaped SnI\(_2\) whiskers.

Fig. 9.5 Growth of one ribbon whisker over the other whisker.

Fig. 9.6(a) X-shaped SnI\(_2\) whiskers.

Fig. 9.6(b) Y-shaped SnI\(_2\) whiskers.

Fig. 9.7 Aggregates of whiskers oriented along one direction.

Fig. 9.8 Branching of the whiskers.

Fig. 9.9 Growth steps on SnI\(_2\) whisker surface.

Fig. 9.10 Striations parallel to [010] direction found on outer surface of SnI\(_2\) whisker.

Fig. 9.11 SEM of striations parallel to <010> found on outer surface of SnI\(_2\) whisker.

Fig. 9.12 SEM of tapering and branching of whiskers.

Fig. 9.13 SEM of projecting end views of solid and hollow whisker crystals.

Fig. 9.14 The typical hollow whisker crystals viewed from the top.
Fig. 9.15  Overgrowth of micro-crystallites oriented in [010] direction on surface of whiskers.

Fig. 9.16  Graph of radius of curvature (R) on the fracture plotted against the whisker thickness (t) for uniform whisker.
Fig. 9.11  X 2000

Fig. 9.12  X 2000

Fig. 9.13  X 1250