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Aging effect on microhardness in NaCl: NaBr mixed crystals

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ABSTRACT

Mixed crystals of NaCl : NaBr with varying composition were grown using Czochralaski technique in an open environments. As-grown crystals were cleaved and microhardness on Vicker's scale were measured on (001) plane in <100> direction at room temperature. It was observed that with an increase in indentation load the hardness first increases and thereafter attains saturation value at higher loads. Moreover, it varies nonlinearly with compositions at all loads irrespective of crystal composition. Crystal of equimolar composition shows maximum hardness than its constituents. These crystals were allowed to remain at room temperature for about a year so as to become aged. It is observed that the aging has no influence on the nature of variation of microhardness number with composition except the fact that the hardness number gets considerably increased. These results were interpreted in terms of an increased resistance to caused by the defect clusters and pinned dislocations surrounded with impurities to the motion of desolations created at the time of indentation.

INTRODUCTION

Hardness is a measure of resistance to permanent deformation or damage. It is an important property of solid state material. It is related to the bond strength as well as defect content of the crystals. So hardness appears to be related to two independent factors; the bond ionicity and the bond polarizability. Andre Julg[1] obtains a relation between these factors as, $Hv = K (1-2/3a^4)$ Where Hv is hardness number on Mohs hardness scale, 'a' is bond ionicity and 'K' is constant determine by the number of rows to which the different atoms belongs in periodic classification of elements. For the microhardness study, the indentation tests are more appropriate. The shape of indentation mark has a close effect on the measurement of the hardness of the given crystal. This test is non–destructive and can provide information up to well inside the surface. The present paper report the effect of aging on microhardness of mixed NaCl: NaBr solid solution over the entire composition range viz 100% NaCl to 100% NaBr, grown by Czochralaski technique.

EXPERIMENTAL

The hardness testing of the material is essential before determining the suitability of any material for a given purpose. The Vickers's pyramid micro indention hardness test provides the study of this mechanical property within a thin surface layer of crystal without any damage to their structure.

Hardness measurements were made with the help of Vickers's pyramid hardness tester attached to Carl-Zeiss universal microscope. Samples of dimension 4mm x 4mm x 3mm were cleaved from the fresh and aged samples of NaCl: NaBr mixed crystals. The aged crystals were grown about two years back by using Czochralaki technique. All the indentations ware performed at room temperature 40^0C and the humidity being 40%. The indentation load varied from 10gm to 50gm. The time of indentation was kept 10 sec. About 8 indentations were made on each sample and average lengths of the diagonal of the marks were measured. The Vicker's hardness has been calculated using the formula

$$Hv = 1.8544 \times P/d^2 \text{ Kg/mm}^2.$$

Where P is load in gm and *d* is diagonal length of indentation mark.

RESULT AND DISCUSSION

The average diagonal lengths of indentation marks were measured and microhardness of these crystals were calculated. The results are shown in graphs. It is clear that at initial state the hardness increases with load approaching towards a constant value at higher loads. At lower loads although the nature of variation of hardness with load is similar for all compositions, the mixed crystals show higher hardness than pure components. However at higher loads the hardness of pure NaCl crystal is more than that of mixed crystal. Whereas the hardness of NaBr crystal is lower at all loads. Moreover, it varies nonlinearly with composition with maximum at around equimolar composition. Hardness is explained in the terms of resistance to the movement of dislocations created during indentation. When indentation is made the fresh dislocations are created and move away from the point of indentation. Higher the density of dislocation in the crystal more would be the resistance encountered by moving dislocation. Shaskolskaya[2] have studied the arrangement of dislocation around indentation mark in alkali halide crystal and concluded that the depth to which dislocations are produced by concentrated stress, are however not of much importance but depends on the indentation load. Verchenya[3] show that hardness increases with decreasing loads in LiF crystals.

Inab[4] established a relation between microhardness and rosette length as $P=bL^n$ Where b is a constant and L is length of rosette. Subba Rao and Hari Babu[5] have suggested that the difference in the ionic radius of the ions constituting the mixed crystals might be responsible for the observed value of microhardness of the mixed crystals. They gave the relation between the hardness number and the inter-ionic distance.

$$H = (\text{con.}).\ r^{-m} + K\ N_a\ N_b$$

Where K is coefficient of hardness, N_a & N_b are respective composition of two components and m is constant. Gerjuzin[6] obtain internal stress of order 10^8 dyne/cm^2 in mixed KCl-KBr crystal whereas in NaCl-NaBr system it was 6×10^8 dyne/cm^2. This provides an evidence for the existence of internal stress in the crystal, which contain ions of different kinds. The origin of such stress is size misfit, which in other words is responsible for increase in hardness when alkali halides form solid solutions[7]. Hardness is mixed crystals could not be related to the energy required to form vacancy pairs in these crystals as suggested by Shukla and Bansigir[8]. Madan[9] has reported that the mixed crystals having nearly equimolar composition shows higher hardness number than their component crystals. The force applied to the pure as well as mixed crystal is higher i.e. a lower number of vacancy pair are created in mixed crystals. In an ideal situation if it is assumed that the force as well as the number of vacancy pair removed remains constant for the pure and mixed crystals, then comparatively more energy is required to form these vacancy pairs in mixed crystals. On the contrary the conductivity measurement shows that the formation energy for a defect is less for mixed crystals. On aging the impurities and other point defect diffuse with in the crystal and get accumulate around dislocation thus these dislocation get pinned. These pinned dislocation act as obstacles in the movement of fresh dislocation created at the time of indentation. Probably that is why on aging the microhardness value gets enhanced. The nature of variation of Hv with composition is observed similar in both set of samples viz. as-grown and aged. This clearly reflects that the basic mechanism, which govern this nature, do not affected by the process of aging.

CONCLUSION

1. Aging increases the microhardness by pinning the dislocations introduced at the time of growth of crystal.

2. Nature of variation of Hv with composition does not change on aging.

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