Brief Reports

Brief Reports are short papers which report on completed research which, while meeting the usual Physical Review standards of scientific quality, does not warrant a regular article. (Addenda to papers previously published in the Physical Review by the same authors are included in Brief Reports.) A Brief Report may be no longer than 3½ printed pages and must be accompanied by an abstract. The same publication schedule as for regular articles is followed, and page proofs are sent to authors.

Formation of concentric rings on the flaked surface of Mylar due to 250-keV H⁺-ion implantation

U. K. Chaturvedi, V. Shrinet,* and A. K. Nigam

Van-de-Graaff Laboratory, Physics Department, Banaras Hindu University, Varanasi-221 005, India

(Received 16 December 1985)

Concentric rings formed on the flaked surface of Mylar after 250-keV H⁺-ion implantation (fluence: 8×10^{15} cm⁻²) have been observed. Unlike those in metallic glasses and in Si single crystals where similar features have been observed due to MeV-energy He⁺ implantation, the number of these rings is very limited. In some cases, several groups of such rings have been observed in a single flaked blister. The cause of formation of such features is discussed in the light of existing theories.

Paszti et al. have observed wavelike features called "pattern formation" in MeV-energy implanted (fluence $\sim 10^{18}$ ions cm^{-2}) amorphous metallic glasses.¹⁻³ Surprisingly, they very recently also observed similar features in silicon single crystals⁴ when they extended their studies to crystalline materials. To theirs as well as our knowledge, no such phenomenon has been reported for polycrystalline metals. This prompted us to look for such features in semicrystalline polymers like Mylar and Kapton also. During the course of our studies on ≥ 100 -keV H⁺-ion implantationinduced blistering in Mylar, we observed the formation of concentric rings on the flaked Mylar surface which is reported here. Although similar to those observed by Paszti et al. for Si single crystals,⁴ the occurrence of these concentric rings in Mylar is not very frequent and does not cover the whole flaked area of an exfoliated blister.

The details of the experimental arrangements have been discussed elsewhere.⁵⁻⁷ After the implantation of 250-keV H⁺ ions up to a fluence of 8×10^{15} cm⁻², which is quite sufficient for flaking of the blisters developed in Mylar, the flaked surface was recoated with a 500-Å-thick Al film so that it would become electrically conducting and not accumulate charge during scanning electron microscopy (SEM). A Philips PSEM-500 was used at 12 keV for these studies.

A typical set of concentric rings formed on the surface of a flaked blister is shown in Fig. 1. The cantilever-type feature shadowing some portions of these rings in the top right corner of the micrograph is debris from the flaked skin. The observed concentric rings are circular in shape, but appear to be elliptical due to the obliqueness of the flaked plane with respect to the electron beam in the SEM. (In the case of normal incidence, the contrast of these rings was not good enough, and it was very difficult to photograph them.) The interspacing of these rings, as measured from the micrograph along the major axis of the ellipse, is dramatically uniform and is 3.6 μ m in Fig. 1. In the case of another flaked blister of the same sample, two sets of such concentric rings lying close to each other are shown in Fig. 2(a). Figure 2(b) is the magnified view of the right lower group of Fig. 2(a). These groups have five and four circular rings, respectively, having approximately a $3-\mu$ m interspacing.

The exact cause of formation of these concentric rings on a flaked surface of Mylar is not very clear at this stage and demands a detailed experimental study. Lovas and Tel⁸ have given a partial theoretical explanation based on diffusion to explain these features on amorphous surfaces. However, on observing similar features in Si single crystals also, Paszti *et al.*⁴ proposed a more general explanation for both amorphous and single crystals by considering a mechanical-stress model instead of a diffusion-based one and have defined a condition which is the critical stress for



FIG. 1. A typical set of four concentric rings observed on the flaked surface of a blister. (Dose: 8×10^{15} H⁺ ions/cm² at 250 keV.) (Bar: 10 μ m.)

33 5834





(b)

FIG. 2. (a) Two sets of concentric rings in another flaked blister and (b) the lower right set of rings at higher magnification. (Dose: 8×10^{15} H⁺ ions/cm² at 250 keV.) [Bar: (a) 10 μ m; (b) 1 μ m.]

*Present address: Polymer Insulation Division, ERDA, Vadodara, India.

- ¹A. Manuaba, F. Paszti, L. Pogany, M. Fried, G. Mezey, T. Lohnes, I. Lovas, L. Pocs, and J. Gyulai, Nucl. Instrum. Methods **199**, 409 (1982).
- ²F. Paszti, M. Fried, L. Pagany, A. Manuba, G. Mezey, E. Kotai, I. Lovas, T. Lohner, and L. Pocs, Nucl. Instrum. Methods 209/210, 273 (1983).
- ³F. Paszti, M. Fried, L. Pogany, A. Manuba, G. Mezey, E. Kotai, I. Lovas, T. Lohner, and L. Pocs, Phys. Rev. B 28, 5688 (1983).
- ⁴F. Paszti, Cs. Hajdu, A. Manuba, N. T. Kotai, L. Pogany, G. Mezey, M. Fried, Gy. Vizkelethy, and J. Gyalai, Nucl. Instrum. Methods Phys. Res. Sect. B 7/8, 371 (1985).

wave formation, i.e., $\sigma_{\text{critical}}^{\omega}$, given by

$$\sigma_{\rm critical}^{\omega} = E[3(1-\nu^2)]^{-1/2} , \qquad (1)$$

where E is the Young's modulus and ν is the Poisson's ratio of the implanted surface. When the flaking takes place before this value of stress (i.e., $\sigma_{\text{critical}}^{\omega}$) is achieved, no wavelike features are expected to occur. On taking the value of E and v for Mylar from the literature,⁹ $\sigma_{\text{critical}}^{\omega}$ comes out to be 3.38×10^9 dyn cm⁻² which is nearly 2.5 times more than that of its UTS (i.e., the maximum stress it can bear before fracture). So, on the basis of this, no such wavelike features should be generated on the implantation-induced flaked Mylar surface. However, since semicrystalline polymers are highly inhomogeneous microscopically in their mechanical properties,¹⁰ regions of higher UTS where Paszti's condition is satisfied may be present at several places within a flaked blister. Thus, at these places such wavelike features may be formed. The probable circular symmetry of UTS variation in these regions gives the exact circular shape to the rings. It appears that as these high UTS regions are not very frequent, only a few sets of these concentric rings are observed in a big flaked blister, and most of the flaked area is not covered by these rings. To understand this interesting phenomenon more thoroughly, a detailed experimental study is needed after which a quantitative theory can be proposed. Such a study is in progress and the results will be reported in due course.

The authors are grateful to the Head of the Department of Metallurgical Engineering, Institute of Technology, Banaras Hindu University, for providing the SEM facility. They gratefully acknowledge financial support given by the Department of Atomic Energy (DAE), Government of India, for this work (Grant No. 37/6/84-G).

- ⁵V. Shrinet, U. K. Chaturvedi, S. K. Agrawal, V. Singh, and A. K. Nigam, Nucl. Instrum. Methods 209/210, 1193 (1983).
- ⁶V. Shrinet, U. K. Chaturvedi, S. K. Agrawal, V. N. Rai, and A. K. Nigam, in *Polyimides*, edited by K. L. Mittal (Plenum, New York, 1984), Vol. 1, pp. 555–571.
- ⁷V. Shrinet, U. K. Chaturvedi, S. K. Agrawal, and A. K. Nigam, Nucl. Instrum. Methods Phys. Res. Sect. B 1, 617 (1984).
- ⁸I. Lovas and T. Tel, Phys. Rev. B 28, 5692 (1983).
- 9V. Shirnet, Ph.D. thesis, Banaras Hindu University, 1985 (unpublished), and references therein.
- ¹⁰R. L. McCullough, in *Treatise on Material Science and Technology*, edited by J. M. Schultz (Academic, New York, 1977), Vol. 10B, pp. 453-540.



FIG. 1. A typical set of four concentric rings observed on the flaked surface of a blister. (Dose: 8×10^{15} H⁺ ions/cm² at 250 keV.) (Bar: 10 μ m.)



(a)



(b)

FIG. 2. (a) Two sets of concentric rings in another flaked blister and (b) the lower right set of rings at higher magnification. (Dose: 8×10^{15} H⁺ ions/cm² at 250 keV.) [Bar: (a) 10 μ m; (b) 1 μ m.]