LASER INDUCED TRANSFORMATION OF THIN AMORPHOUS CrNi FILMS

M. Bîrjega, M. Dinescu, C. Sârbu, M. Chvoika

► To cite this version:

M. Bîrjega, M. Dinescu, C. Sârbu, M. Chvoika. LASER INDUCED TRANSFORMATION OF THIN AMORPHOUS CrNi FILMS. Journal de Physique IV Proceedings, 1991, 01 (C7), pp.C7-73-C7-76. 10.1051/jp4:1991716 . jpa-00250836

HAL Id: jpa-00250836

<https://hal.science/jpa-00250836>

Submitted on 1 Jan 1991

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

LASER INDUCED TRANSFORMATION OF THIN AMORPHOUS CrNi FILMS

M.I. BÎRJEGA, M. DINESCU, C. SÂRBU and M. CHVOIKA*

*Institute of Atomic Physics, P.O. Box MG-6 Magurele, R-76900 Bucharest, Romania***Institute of Physics, Na Slovance 2, 180.40, Praha 8, Czechoslovakia*

Abstract. TEM and TED are presented on the crystallization and oxidation of the 60 nm thick amorphous CrNi (55:45) films supported on electron microscope copper grids when submitted in air to the action of a single photodissociation iodine laser ($\lambda \approx 1.315 \mu\text{m}$) pulse. The laser could operate in Q-switched regime, with a typical fullwidth half-maximum τ_{FWHM} of 5 ns and a power intensity I , of $6.7 \times 10^6 \text{ W}\cdot\text{cm}^{-2}$.

1. Introduction

The structural and chemical transformation in amorphous CrNi (55:45) and Cr thin films supported on EM copper grids induced by air irradiation with a single-pulse TE CO₂ laser ($\lambda = 10.6 \mu\text{m}$; $\tau = 2.5 \mu\text{s}$; $I = 0.3 \div 1.2 \times 10^6 \text{ W}\cdot\text{cm}^{-2}$), a free running ruby laser ($\lambda = 0.69 \mu\text{m}$; $\tau = 400 \mu\text{s}$; $I = 7.5 \times 10^2 \text{ W}\cdot\text{cm}^{-2} \div 13.7 \times 10^3 \text{ W}\cdot\text{cm}^{-2}$), a Q-switch ruby laser ($\lambda = 0.69 \mu\text{m}$; $\tau = 60 \text{ ns}$, $I = 0.7 \times 10^5 \text{ W}\cdot\text{cm}^{-2} \div 0.9 \times 10^6 \text{ W}\cdot\text{cm}^{-2}$), an excimer laser ($\lambda = 0.308 \mu\text{m}$; $\tau = 20 \text{ ns}$; $I = 0.6 \times 10^5 \text{ W}\cdot\text{cm}^{-2} \div 1.2 \times 10^6 \text{ W}\cdot\text{cm}^{-2}$) was previously studied by us [1 - 5] using TEM and TED. In all the performed experiment we observed that the EM copper grid meshes ($\phi \approx 120 \mu\text{m}$), which support the CrNi (55:45) or Cr thin film, channel the incident laser beam so that at mesh level in the irradiated spot [$0.5 \times 10^{-2} \text{ cm}^2 \div 7 \times 10^{-2} \text{ cm}^2$] temperature gradients develop from the center of the mesh towards its borders. Thus at grid mesh level in the irradiated area, a sequence of microstructures could be noticed beginning near the grid mesh with amorphous untransformed CrNi (55:45) or Cr, then fine crystallized CrNi or Cr, with a microstructure similar to that obtained by vacuum annealing treatment [6] or vacuum deposition at higher substrate temperatures [7, 8]. Perforation, melting and cracking are characteristic to the centre of the mesh, where the oxidation and crystallization processes attain the highest degree, especially at the Cr thin films [4, 5].

2. Experimental procedure

Icosahedral amorphous [9] 60 nm thick CrNi (55:45) films were deposited onto freshly air-cleaved KCl substrates using a standard RF sputtering apparatus. Details on the deposition conditions are given in our previous paper [6]. The films were removed from the KCl sub -

strates by immersion in distilled water and floated off onto EM copper grids (i.e. networks of circular 120 μm diameter holes). These films are referred to as free standing (FS) samples [10]. The irradiation was performed in air, at room temperature, using a Q-switch ($\tau_{\text{FWHM}} = 5 \text{ ns}$), photodissociation iodine laser ($\lambda = 1.315 \mu\text{m}$). The laser beam was focused at normal incidence onto the FS, CrNi(55:45) samples, ensuring a intensity of $6.7 \times 10^6 \text{ W}\cdot\text{cm}^{-2}$ in the irradiation spot area ($0.5 \times 10^{-2} \text{ cm}^2$). The irradiation effects were observed by examination of the free-standing (FS) CrNi (55:45) samples in a TEM 200 CX electron microscope working at 200 kV in the conventional TEM and TED modes.

3. Results and discussions

The TEM and TED investigations performed on the photodissociation iodine laser ($\lambda \approx 1.315 \mu\text{m}$, $\tau_{\text{FWHM}} = 5 \text{ ns}$, $I = 6.7 \times 10^6 \text{ W}\cdot\text{cm}^{-2}$) irradiated amorphous CrNi (55:45) FS thin films have essentially confirmed our previous results [1 - 5]

The Role of EM Copper Grids in the Crystallization Process of the CrNi (55:45) Thin Films

Despite the large area of the irradiation spot ($0.5 \times 10^{-2} \text{ cm}^2$) and the uniformity of the incident energy, the effects of the 5 ns single laser pulse are clearly a characteristic of the corresponding area of one mesh ($\sim 7.8 \times 10^3 \mu\text{m}^2$) of the EM copper grid. As we can see from the electronmicrographs (fig. 1, low - magnification and enlarged view) like in our previous experiments [1 - 5], under the action of the laser irradiation, we observed the formation of four zones with characteristic microstructures denoted by A, B, C and D. The perforation, melting or cracking of the CrNi (55:45) thin film (zone A) are induced in the centre of the copper grid mesh by the heat release into the 60 nm thick sample and/or the stresses produced by their sudden crystallization and oxidation (fig. 1).

Surrounding zone A, there is zone B where the melted superficial layer is rippled with periodicities λ ranging from $0.58 \mu\text{m}$ to $1 \mu\text{m}$. Of less extent at low magnification, we find zone C located next to B. Electronmicrograph and electron diffraction patterns from this zone (C_1, C_2) certify that the temperatures attained are stepwise lower so that, zone C_2 , marks the beginning of a heterogeneous crystallization by the nucleation of the metastable extended γ -NiCr (fcc) crystals. In zone C_1 higher temperatures are to be attained because, both the EM and the ED data give evidence that the γ -NiCr crystals are grown in size and also that small crystals of the Cr_3O_4 oxide are formed. Zone D corresponds to the thin film next to the mesh edge that maintains its original structure [6].

As we have mentioned from the beginning the formation of such a microstructure range can only be accounted for by a local laser induced temperature distribution $T(r, t)$, which is essentially generated by heat losses to the copper grid support. If as a rule, due to the appearance of these thermal gradients between the centre and the mesh edge of the EM copper grid we find the mentioned A, B, C and D microstructures in the irradiated area of the amorphous CrNi(55:45) or Cr thin films independently of the laser parameters and deposited energy [1 - 5] some differences can be noticed in the crystallization or oxidation behaviour. So at the lowest intensity ($I = 1.4 \times 10^2 \text{ W}\cdot\text{cm}^{-2}$) at the CrNi (55:45) one pulse irradiated ($\lambda = 0.69 \mu\text{m}$, $\tau = 400 \mu\text{s}$) the first crystallization step (zone C_2) is the α -CrNi extended solid solution [1] obtained by a polymorphous type of crystallization. Chaplanov

Fig. 1. Electronmicrographs of one EM copper grid mesh from the laser irradiated ($\lambda = 1.315 \mu\text{m}$; $\tau_{\text{FWHM}} = 5 \text{ ns}$; $I = 6.7 \times 10^6 \text{ W} \cdot \text{cm}^{-2}$) area ($0.5 \times 10^{-2} \text{ cm}^2$) of the (FS) CrNi (55 : 45) film and their corresponding enlarged view. The four A, B, C₁, C₂ [1 - 5] zones are marked on the electronmicrograph and the corresponding electron diffraction patterns. Indices hkl of the indexed phases :
 (*) $\gamma\text{-NiCr}$ [ASTM-4-0850] ; (o) Cr_3O_4 [ASTM-12-559] ;
 (Δ) $\alpha\text{-Cr}_2\text{O}_3$ [ASTM-6-0505] ; (**) NiCr_2O_4 [ASTM-4-0763].
 Ripple periodicities A ranging from $0.58 \mu\text{m}$ to $1 \mu\text{m}$.

and Tochitsky [12] observed in the case of one pulse irradiated polycrystalline textured 100 to 500 nm thick Cr film that the formation of the Cr_3O_4 oxides is favored both by an increase in the power density from $6 \times 10^4 \text{ W.cm}^{-2}$ to $1.3 \times 10^5 \text{ W.cm}^{-2}$ and by using vacuum instead of air. We also observed, a laser power density and pulse duration dependence in the Cr_3O_4 oxide formation and crystallization in air irradiated thin amorphous Cr and CrNi (55:45) films [4, 5].

4. Conclusions

Lateral differences in the crystallization and oxidation of 60 nm thick amorphous CrNi (55:45) films supported on EM copper grids subjected in air to the action of a single laser pulse 5 ns in width (FWHM) delivered from a photodissociation iodine laser ($\lambda \approx 1.315 \mu\text{m}$) at power density of $6.7 \times 10^6 \text{ W.cm}^{-2}$ are revealed by TEM and TED investigations.

References

- [1] M.I.Birjega, R.M.Birjega, C.A.Constantin, M.Dinescu, I.Th.Florescu, I.N.Mihăilescu, N.Popescu-Pogrion and C.Sârbu, Thin Solid Films, 145 (1986) 111.
- [2] I.Ursu, M.I.Birjega, M.Dinescu, I.N.Mihăilescu, N.Popescu-Pogrion, C.A.Constantin, I.Ketskemety and E.Szill, MRS Proc. Vol. 74 (1986) 197.
- [3] I.Ursu, M.I.Birjega, C.A.Constantin, M.Dinescu, I.N.Mihăilescu, N.Popescu-Pogrion, I.Ketskemety and E.Szill, Supplement à la Revue Le Vide, Les Couches Minces 235 (1987) 573.
- [4] I.Ursu, M.I.Birjega, M.Dinescu, I.N.Mihăilescu, N.Popescu-Pogrion, L.Ribco, I.Ketskemety, L.Nanai and E.Szill, Rev.Roum.Phys. 34 (1989) 195.
- [5] I.Ursu, M.I.Birjega, M.Dinescu, I.N.Mihăilescu, N.Popescu-Pogrion, L.Ribco, A.M.Prokhorov, V.I.Konov and V.N.Tokarev, Applied Surface Science 36 (1989) 640.
- [6] M.I.Birjega, C.A.Constantin, I.Th.Florescu and C.Sârbu, Thin Solid Films 92 (1982) 315.
- [7] C.Sârbu, S.A.Rău, N.Popescu-Pogrion and M.I.Birjega, Thin Solid Films 28 (1975) 311.
- [8] L.Lassak and K.Hieber, Thin Solid Films 17 (1973) 105.
- [9] M.I.Birjega and N.Popescu-Pogrion, Thin Solid Films 171 (1989) 33.
- [10] R.Andrew, L.Baufay, A.Pigeolet and L.D.Laude, J. Appl. Phys. 53 (1982) 4862.
- [11] M.I.Birjega, C.A.Constantin, I.Th.Florescu, N.Popescu-Pogrion and C.Sârbu, Rev. Roum. Phys. 29 (1984) 219.
- [12] A.M.Chaplanov and E.I.Tochitsky, Thin Solid Films 116 (1984) 117.