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Citation: Applied Physics Letters **98**, 094103 (2011); doi: 10.1063/1.3560053
View online: http://dx.doi.org/10.1063/1.3560053
View Table of Contents: http://scitation.aip.org/content/aip/journal/apl/98/9?ver=pdfcov
Published by the AIP Publishing

APPLIED PHYSICS LETTERS **98**, 094103 (2011)

Characterization of write-once blu-ray disk containing Cu–Al/Si recording layer using transmission electron microscopy

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(Received 7 January 2011; accepted 1 February 2011; published online 1 March 2011)

Microstructure change in write-once blu-ray disk containing Cu–Al/Si recording layer was investigated by transmission electron microscopy. Nanoscale crystallites were found to comprise of the Cu–Al/Si recording layer before and after signal writing and the energy dispersive spectroscopy revealed insignificant composition fluctuation in disk sample. Analytical results indicated the signal properties of disk samples are correlated with a moderate improvement of crystallinity and the formation of Cu and Si solid-solution phases due to element mixing in mark area, rather than the formation of Cu_3Si silicide and recrystallization of recording layer as reported by previous studies. © *2011 American Institute of Physics*. [doi:10.1063/1.3560053]

As a popular high-speed recording medium for write-once blu-ray disk (BD-R), the Cu/Si bilayer has been widely studied for its microstructure and correlation to signal properties.[1–9] Various tools, namely, Auger electron spectroscopy (AES),[3,4] transmission electron microscopy (TEM),[6] phase-change kinetics of thin films,[7,8] and conductive atomic force microscopy (C-AFM),[9] have been employed to analyze the operative mechanism of Cu/Si-based BD-R disk samples. AES revealed a diffusion of Si into Cu and a formation of CuSi alloy containing 25–30 at. % Si during the laser writing.[3] Though AES is a valuable tool to gain diffusion information in Cu/Si couple, it can only extract the data from a totally initialized disk rather than a real disk due to the limitation of spatial resolution. Russell *et al.*[6] and Her and Wu[7] separately observed the formation of Cu_3Si silicide and recrystallization of amorphous Si (*a*-Si) in *a*-Si/Cu sample and the kinetic studies[7,8] identified the phase transition modes of Cu/Si system. Nevertheless, these works were performed in the static manner within relatively slow heating rates and the results are unlikely applied to the real disk subjected to an ultrafast laser heating. Mank *et al.*[9] demonstrated that C-AFM can detect the recording bits in various types of optical disks by taking the advantage of electrical conductivity difference in mark and nonmark areas; it nevertheless is an indirect microstructure characterization and offers no composition information in the disk samples.

TEM is a powerful tool to characterize the microstructure of optical disks[10,11] and, with the aid of energy dispersive spectroscopy (EDS), it can also distinguish the composition change in small scale. By using the hot-stage TEM, Russell *et al.* observed a dendrite-type structure and Cu_3Si phase in the crystallization of Cu/Si layer.[6] However, the analysis was done on a Cu/Si thin-film diffusion couple, not a Cu/Si TEM sample based on a real BD-R disk. Due to the difficulty of TEM sample preparation,[9] a direct TEM observation on Cu/Si-based BD-R optical disks is still missing and, thus, the correlation of signal properties to microstructure change in recording layer remains unclear. This work reports the evolution of microstructure and composition in the Cu–Al/Si BD-R disk samples characterized by TEM/EDS. According to the experimental findings, the operative mechanism of Cu/Si-based BD-R disks is discussed.

The optical disk samples were prepared in accord with the BD specifications[12] using a commercial sputtering system at a background pressure $\leq 10^{-6}$ torr. The multilayer structure of disk sample is: polycarbonate substrate (1.1 mm)/Ag reflection layer (80 nm)/$ZnS-SiO_2$ (30 nm) dielectric layer/Cu–Al (5 nm)/Si (5 nm)/$ZnS-SiO_2$ (30 nm) dielectric layer/cover layer (0.1 mm). A small amount of Al added in Cu layer is to modulate the recrystallization temperature of Cu/Si system (=485 °C) (Refs. 6–8) and its effect on the signal properties and microstructure of disk samples is also evaluated.

The disk samples recorded at the optimized condition were established by measuring the signal properties including the jitter value, symbol error rate (SER), modulations and optimum write power (P_w) of the BD-R samples using a dynamic tester (ODU-1000, PULSTEC) within the conditions listed in Table I.[12] The optimum test results at 2× and 4× recordings are presented in Table II and the eye patterns

TABLE I. Dynamic test conditions for BD-R disk samples.

Disk diameter (mm)	120	
User capacity (Gbyte)	25	
Substrate thickness (mm)	1.1	
Cover layer thickness (mm)	0.1	
Wavelength (nm)	405	
Numerical aperture (NA)	0.85	
Track pitch (μm)	0.32	
Minimum mark length (nm)	149	
Modulation code	run-length-limited (RLL) (1,7)	
Equalizer	Limit equalizer	
Recording speed	2×	4×
Channel clock (MHz)	132	264
User Data transfer rate (Mbps/s)	72	144
Liner velocity (m/s)	9.84	19.68

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TABLE II. Optimum test results for disk samples subjected to 2× and 4× recordings at multitracks condition.

Recording speed	2×	4×
P_w (mW)	6	7.9
Jitter (%)	6.2	6.3
SER	3×10^{-5}	1.8×10^{-5}
Modulation	0.6	0.6

corresponding to random signal recording read directly from the oscilloscope at 2× and 4× recordings are separately showed in Figs. 1(a) and 1(b). This illustrates the random signals ranging from 2 T to 8 T could be well written in the Cu–Al/Si BD-R disks and, consequently, these samples were transferred to an JEOL FX-II 2010 TEM for microstructure and composition characterizations. The plan-view TEM samples were prepared according to the method reported by Chen *et al.*[13] Note that an accurate control of ion milling condition to remove the Ag reflection layer of disk sample is crucial to the TEM sample preparation.

Representative TEM images for disk samples subjected to 2× and 4× recordings are separately presented in Figs. 2(a) and 2(b). Signal marks with various recording lengths residing in the grooves of disks can be readily seen. The randomly recorded marks exhibit sharp edges and their shapes are not affected by the change of recording speed. The bit length is about 150 nm for 2T signal as depicted in Fig. 2, in agreement with the smallest mark length defined by the BD-R specifications.[12] Hence, Cu–Al/Si system is a promising recording medium for high-speed BD-R products.

Figure 3(a) depicts the locations subjected to EDS (Link ISIS 300) analysis and the chemical compositions corresponding to these locations are plotted in Fig. 3(b). As shown in Fig. 3(b), there is no obvious composition fluctuation when the detection shifts from nonmark area to mark area. The uniform composition distribution clearly implies the absence of silicide phases such as Cu_3Si in mark area. This is ascribed to the ultrafast heating/cooling feature of laser writing which inhibits the long-range atomic diffusion for silicide formation and recrystallization during signal recording.

The TEM image of disk sample shown in Fig. 4(a) delineates the selected area electron diffraction (SAED) patterns taken from the mark (the inset at lower left-hand corner) and nonmark (the inset at upper right-hand corner) areas. Instead of a single diffuse ring, the broad concentric diffraction rings in both SAED patterns indicates the recording layers are comprised of the nanoscale crystallites regardless of the signal recording. As a matter of fact, the fine granulates about several nanometers in sizes in mark area can be visibly seen in the high-magnification insets at lower right-hand corners of Figs. 2(a) and 2(b) [also see Figs. 3(a) and 4(a)]. The lack of long-range crystallinity in Cu–Al/Si layer hence results in the broadening of diffraction peaks as predicted by the Scherrer's formula.[14]

A careful examination on SAED patterns found that the diffraction rings of mark area exhibit some nonuniformity in intensity distribution in comparison with those of nonmark area. Blur, bright spots decorated in the broad diffraction rings can be observed in an enlarged SAED pattern of the mark as shown in Fig. 4(b), implying a moderate crystallinity

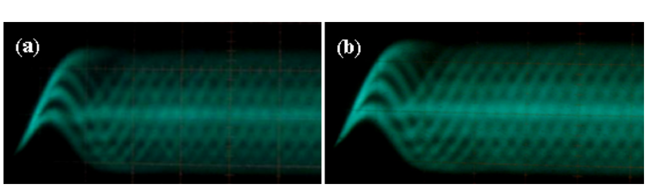

FIG. 1. (Color online) Eye patterns of random signals read directly from oscilloscope for disk samples subjected to (a) 2× and (b) 4× recordings at multitracks condition.

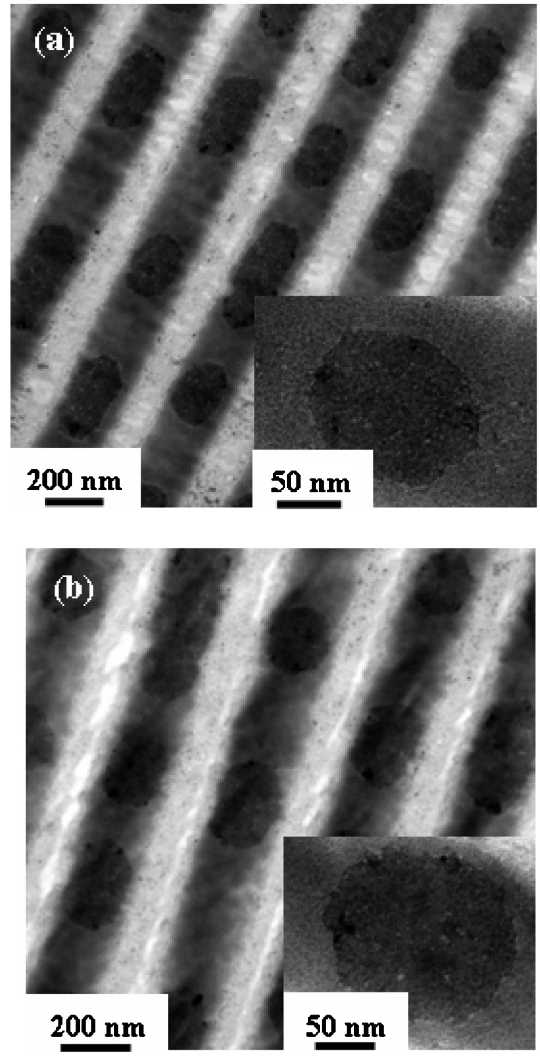

FIG. 2. TEM micrographs of recorded BD-R disk samples subjected to (a) 2× and (b) 4× recordings.

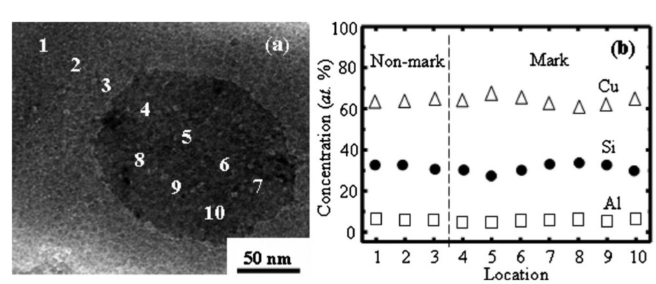

FIG. 3. (a) Locations of EDS analysis of a signal mark and it vicinity and (b) the variations in element compositions deduced from the EDS analysis on the nonmark and mark areas.

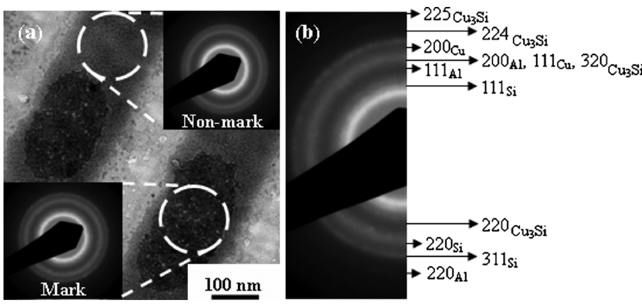

FIG. 4. (a) SAED patterns taken from the nonmark (upper right-hand corner) and mark (lower left-hand corner) areas of disk sample. The locations subjected to diffraction analysis are indicated by broken circles. (b) The indexing of SAED patterns taken from marks area.

improvement induced by laser heating. The melting point of Si is 1414 °C while the Cu–Al alloy utilized in this study melts at about 1045 °C. Furthermore, thermal simulation studies of optical disks[15–18] indicated the disk sample is unlikely heated over 800 °C at P_w=7.9 mW for the 4× recording performed in this work. Instead of melting induced by laser irradiation, the Cu–Al/Si layer should thus experience a heating process similar to the rapid thermal annealing. Since the writing process was completed in an extremely short time span, the preliminary stages of annealing, e.g., recovery via subgrain motions and polygonization,[19] would dominate the structure rearrangement. Later stage of annealing, i.e., recrystallization involving the nucleation and grain growth, is negligible since previous TEM analysis has revealed no obvious change in grain morphology of mark area.

The SAED pattern of mark area was indexed with the aid of the Joint Committee of Powder Diffraction Standards (JCPDS) for Cu, Si, and Cu_3Si phases (JCPDS file card No. 04-0836, 27-1402, and 23-0224). Figure 4(b) iterates the absence of Cu_3Si silicide phase in the mark since no match could be found for Cu_3Si phase and, in conjunction with the previously presented TEM/EDS results, the mark should be comprised of the nanoscale Cu and Si solid-solution phases. Formation of Cu-rich solid-solution phase can be deduced from the study regarding of Si diffusion into Cu (Ref. 3) and the high solubility of Si in Cu (e.g., >11 at. % at about 1100 K) in accord with the Cu–Si phase diagram.[20] On the other hand, the solubility of Cu in Si is relatively low as indicated by the Cu–Si phase diagram. However, diffusion length ($\sqrt{Dt}$) is about 12.7 nm at t=50 ns (i.e., the time duration for short T signal recording) for Cu interstitial diffusion in Si at 1000 K (=727 °C),[21] a value greater than the thickness of Si layer in disk sample. Furthermore, numerous grain boundaries presenting in between the nanoscale Cu and Si grains might serve as the fast diffusion paths. Hence, in addition to the structure rearrangement, laser heating might also cause the elements mixing and result in the Cu- and Si-rich solid-solution phases in the recording layer. According to above findings, the operative mechanism in Cu–Al/Si BD-R disk is ascribed to a moderate improvement of crystallinity and the formation of solid-solution phases in mark area which, in turn, lead to the difference in optical reflectivity and the satisfied signal properties presented above.

In summary, this study demonstrates a direct TEM characterization on the BD-R disk containing Cu–Al/Si recording layer. In the disk sample with the optimized structure and writing strategy, EDS analysis revealed the signal recording causes insignificant composition fluctuation and, in conjunction with the SAED analysis, the formation of Cu_3Si silicide phase and recrystallization of recording layer during signal writing are excluded. In accord with the TEM findings, the knowledge of diffusion and the thermal simulation of optical disks reported previously, signal properties of Cu–Al/Si BD-R disk samples are correlated with a moderate improvement of crystallinity via the preliminary structure recovery processes and the formation of Cu- and Si-rich solid-solution phases due to the element mixing in mark area. Analytical results presented above greatly benefit the clarification of operative mechanism in Cu/Si-based BD-R disks.

This work is support by the National Science Council (NSC), Taiwan, R.O.C., under the Contract No. NSC98-2221-E-009-037-MY2.

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