Proceedings of SPIE—The International Society for Optical Engineering

Volume 695

Optical Mass Data Storage II

Robert P. Freese, Maarten DeHaan, Albert A. Jamberdino Chairs/Editors

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18-22 August 1986 San Diego, California

Published by
SPIE—The International Society for Optical Engineering
P.O. Box 10, Bellingham, Washington 98227-0010 USA
Telephone 206/676-3290 (Pacific Time) • Telex 46-7053

SPIE (The Society of Photo-Optical Instrumentation Engineers) is a nonprofit society dedicated to advancing engineering and scientific applications of optical, electro-optical, and optoelectronic instrumentation, systems, and technology.

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Please use the following format to cite material from this book:

Author(s), "Title of Paper," Optical Mass Data Storage II, Robert P. Freese, Maarten DeHaan, Albert A. Jamberdino, Editors, Proc. SPIE 695, page numbers (1986).

Library of Congress Catalog Card No. 86-62602 ISBN 0-89252-730-7

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Printed in the United States of America.

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Conference 695, Optical Mass Data Storage II, represented a one-conference program on Optical Mass Data Storage held at SPIE's 30th Annual International Technical Symposium on Optical and Optoelectronic Applied Sciences and Engineering.

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Volume 695

INTRODUCTION

The year of 1986 represented exciting times for optical storage technology. Commercial optical storage vendors appeared to be overcoming earlier media problems and began shipping in volume. Digital audio disks literally took the world by storm, placing optical disk systems in more people's living rooms at lower cost than ever imagined a few short years ago. Discussion of a wide variety of government and commercial applications became widespread, with focus shifting from the feasibility of the baseline technology to the issues involved in systems integration and manipulation of data bases not achieved before in real applications.

The SPIE 1986 Optical Mass Data Storage Conference was an exciting meeting that reflected these changes in the external environment. It consisted of five days of meetings covering erasable and nonerasable media, systems and subsystems, systems integration, and commercial and government applications. The 61 papers presented represented only a part of the excitement of this conference, as the personal interactions and hall discussions among the over 300 attendees were equally important. The technical focus of the conference showed more and more emphasis on manufacturing issues encountered in production of media and systems and on systems integration issues. This trend will probably continue in the future with more and more papers on the actual application of optical storage systems to real problems. In the baseline technology area, magneto-optical disks appeared to accelerate in interest and achievability.

We in the optical storage business should be proud of the technical and organizational accomplishments represented by the trends we see. The area of optical mass data storage has truly moved from a gleam in the eye to a technology satisfying a real customer need for a better way to handle today's growing information needs. The technology developments are far from finished, but a technology base has been established that provides the foundation for data storage for years to come.

Robert A. Sprague
Xerox/Palo Alto Research Center

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Session 1

Nonerasable Media

Chair
Robert P. Freese
3M Company

New write-once media based on Te-TeO2 for optical disks

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Matsushita Electric Industrial Co., Ltd.

3-15 Yagumo-Nakamachi, Moriguchi, 570, Japan.

Abstract

Thin film media composed of Te-TeO2 (TeOx) containing Pd additive was found to have a feasibility of being applied to high reliability data file optical disks. The carrier to noise ratio (C.N.R.) exceeds 56 dB at 1,200 r.p.m. disk rotation.

The response time of saturation amplitude after a pulsed laser power irradiation was less

than 300 nsec and is able to apply to high speed read verify systems.

From acceleration environmental stress test results, no defect variation was observed. By the Arrhenius plot method, the disk life has been estimated to be more than 10 years at the storage environment 32 degree C, 80 %RH. The recording and degradation mechanism will be also discussed.

1. Introduction

The optical recording disk is expected to be applied to computer memory because of its

high density and archival storage characteristics (1),(2).

Since 1972, we have investigated optical recording media, and sub-oxide thin films have been found to have feasibility in applying to optical recording disks, such as TeOx, GeOx, SbOx and InOx etc.(3). Among them, Te-TeO2 (TeOx) film shows excellent characteristics (4) of recording sensitivity and stability compared to pure Te film media and has been applied to video and document file disks.

On Te-TeO2 (TeOx) film media, some transient phenomena has been observed, which shows the

amplitude and C.N.R. increasing after a pulsed laser irradiation.

In an attempt to produce short transient time film media, we found that Palladium additive to Te-TeO2 (TeOx) was effective in eliminating this phenomena.

2. Disk preparation

A thin film active layer was deposited on a disk substrate by an electron beam vacuum evaporation method. The materials were evaporated from two different sources. One was Te-TeO2 material source, and the other was Pd source. By the two source evaporation method, on the disk substrate, thin films composed of Te, TeO2 and Pd elements was deposited.

A typical deposition rate was 2 nm/sec and the thickness of the optical active layer was around 150 nm. The disk substrate used was an injection molded polycarbonate plastic with

thickness of 1.18 mm, and of diameter of 130 mm.

On the surface side, laser guide tracks were pregrooved. The track geometry was a 1.65

um track pitch, a 0.8 um track width and $\lambda/8n = 65$ nm track depth.

After the active layer was deposited on the disk substrate, Epoxy photo polymerizing adhesive resin was spread over the film and then a protective plastic plate similar to the disk substrate was on to the film, resulting in so called solid structure, compared to the air-sandwich structure.

The simple disk structure was composed of a pregrooved polycarbonate substrate, active layer, above it an adhesive resin layer and finaly, a bonded protective polycarbonate substrate plate

Polycarbonate plastic resin was chosen for its thermal stability. Its high distortion temperature is nearly equal to 145 degree C and higher than that of PMMA.

3. Dynamic performance of disks

3-1. Disk sensitivity

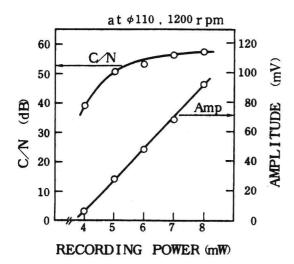
Disk sensitivity was measured by recording and reading signals at various laser recording power levels.

Fig.1 shows a typical C.N.R. and an amplitude vs. recording power characteristics.

The measurement condition was that the carrier frequency is 2 MHz, 50 % duty cycle using laser diode with wave length of 830 nm and an objective lens with N.A. of 0.5 and a disk rotation speed of 1,200 r.p.m..

The amplitude of the recorded signal increased as the recording power increased, and C.N.R. were measured by a spectrum analyzer with a resolution band width of 30 KHz.

Fig. 2 shows a typical frequency characteristics of the disk. More than 50 dB C.N.R. was obtained at 4 MHz at outer tracks of diameter 110 mm and at 2 MHz at inner tracks of diameter 65 mm, respectively. In these cases the recorded dot length was calculated to be 0.85 um.



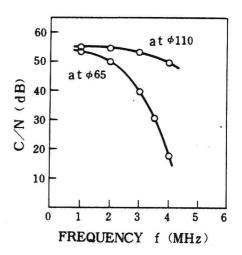


Figure 1. The measured characteristics of the carrier to noise ratio and amplitude vs. recording power.

Figure 2. Typical frequency characteristics of the disk

3-2. Rapid response characteristics

Te-TeO2 (TeOx) active layer, previously reported optical recording media, shows high sensitivity characteristics such as, high C.N.R. of more than 55 dB at 1,800 r.p.m. and more than 5 MHz at the track position of diameter 190 mm (5).

And it was observed some transient phenomena of amplitude increasing after a pulsed laser irradiation.

Fig.3(a) shows the amplitude increasing phenomena on Te-TeO2 (TeOx) active layer after the laser irradiation. From 300 nsec to 66 msec, the amplitude increases to 1.3 times.

The transient time measurement procedure is using 2 beam optical head, one is for recording, and the other is for reading. distance between the two beam spots is 2.3 um and at disk revolution at 900 r.p.m. at an diameter of 165 mm track position of sample disk having diameter 200 mm. The reading spot can read the recorded signal immediately, 300 nsec after the recording pulsed laser irradiation. The second measurement was done time is after one disk revolution at 900 r.p.m.. Successive measurement was carried out by the interval of 66 msec.

For Pd added Te-TeO2 (TeOx) active layer disk, the recorded signal amplitude variation is shown in Fig. 3(b).

The amplitude level difference after 300 nsec and after one revolution of 66 msec is almost the zero.

By this measurement the amplitude transient phenomena was found to be eliminated by the addition of Pd to the Te-TeO2 (TeOx) active layer.

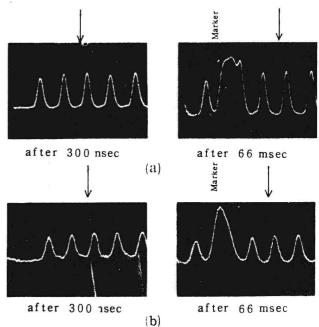


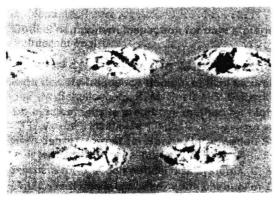
Figure 3. Amplitude variation after the pulsed laser power irradiation.

- (a) Te-TeO2 (TeOx) active layer
- (b) Pd added Te-TeO2 (TeOx) active layer

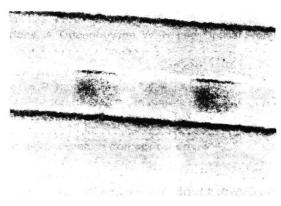
Fig.4(a),(b) shows T.E.M. (transmission electron microscope) observation results of recorded dots on these active layers. For the Te-TeO2 (TeOx) active layer, the recorded dots contain large grains, such as observed in Fig.4(a). In contrast to this figure, Fig.4(b) shows Pd added Te-TeO2 (TeOx) recording dot, where only small crystalline grains are observed.

In case of Te-TeO2 (TeOx) active layers, an electron diffraction analysis showned only a hallo pattern from the unrecorded area, and from the recorded dot, Te crystalline diffraction spot.

In case of Pd added Te-TeO2 (TeOx). the diffraction pattern from the unrecorded area was an uniform ring, and from the recorded dot, PdTe2 crystalline spots appeared in the diffraction ring pattern.



(a)



(b)

Figure 4. Photograph of dots recorded by pulsed laser irradiation, measured by transmission electron microscopy (T.E.M.) (a) Te-TeO2 (TeOx) active layer (b) Pd added Te-TeO2 (TeOx) active layer

4. Disk life

One of potential applications of optical recording is archival mass storage. And the environmental stability of the recording media is of interest.

We evaluated the degradation of the disk using Pd added Te-TeO2 (TeOx) active layer. To obtain some information concerning long-term storage behavior, we have used accelerated stress testing. The characteristics of the recording media were measured as a function of storage time under various temperatures, in both dry (<8 %RH) and high humidity (80 %RH) conditions.

Fxtrapolation of the data to specified environmental conditions, provide us with a plausible estimate of the behavior of the media under long term storage (6).

The temperature dependence of the thermal and the chemical reactions, which are known to be responsible for degradation, is as an exponential relation with temperature such as the Arrhenius plot method.

Acceleration test procedure

Acceleration stress conditions are for example 100 degree C dry (< 8 %RH), 90 degree C, dry (< 8 %RH) and 90 degree C, 80 %RH and 80 degree C, 80 %RH, respectively.

Disks are stored in the test chamber without any disk protection covers such as cartridges. To avoid disk destruction, such as crushing or peeling off and condensation of vapor on the disk surfaces, the pull out condition of the disk from the test chamber should be restricted at a rate within 10 degree C/hour and 10 %RH/hour.

Measurement parameters and results

In this paper, we will discuss various criteria those are commonly used for measuring the life of optical recording media (7).

The first is carrier to noise ratio (C.N.R.) and the second is defect number or defect length ratio named defect-originated error rate (D.E.R.) which is the more important parameter. The third is bit error rate (B.E.R.) of the most important parameter.

The optical disk life characteristics are of two types. The first corresponds to storage data life included in reading characteristics (Archival life) and the second corresponds to writable data life, included in recording characteristics (Shelf life). Both life characteristics of the disk using Pd added Te-TeO2 (TeOx) active layer were measured.

Fig.5(a),(b) and (c),(d) show archival and shelf C.N.R. data. The dry condition and (b), 80 %RH condition is (c) and (d), respectively. In the dry condition, at 100 degree C, after 1500 hours, writable (Shelf) C.N.R. degraded about -3 dB, and at 90 degree C -3 dB degraded, occurred after more than 7000 hours. The archival C.N.R. variation is small compared to the shelf C.N.R. variation.

These results showed that the degradation of Pd added Te-TeO2 (TeOx) active layer was writable sensitivity change.

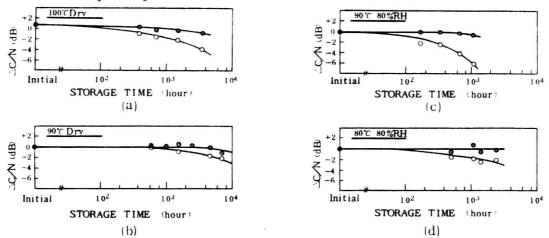


Figure 5. Acceleration test results of reading C/N (Archival) and writable C/N (Shelf) as a function of storage time.

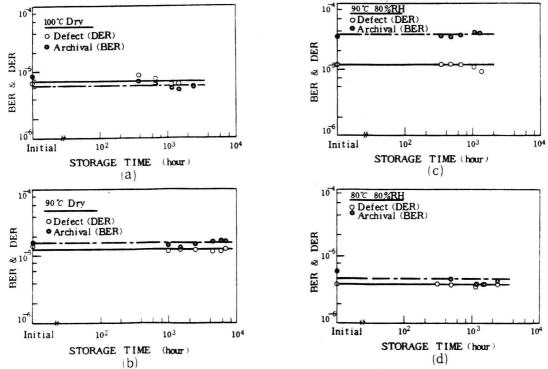
; Archival C/N 0

(a) 100 degree C, dry (<8%RH) (b) 90 degree C, dry (<8%RH)

; Shelf C/N (c) 90 degree C, 80 %RH (d) 80 degree C, 80 %RH

Figs.6(a) (b) and (c),(d) show the defect variation and the B.E.R. variation of initially recorded data so called archival B.E.R. data. Dry conditions are (a) and (b) and 80 %RH conditions are (c) and (d), respectively. The definition of D.E.R. was that, counting the summation of the pulse width of amplitude originated from any defects, and divided by measured time corresponding to measured track length.

The amplitudes counted were those greater than fixed the threshold level. Both defect (D.E.R.) and recorded signal raw B.E.R. show almost no change.



Acceleration test results of defect variation (D.E.R.) and reading raw bit error rate (Archival B.E.R.) as a function of storage time.

(); Defect D.E.R @ : Archival B.E.R.

(a) 100 degree C, dry (<8%RH) (b) 90 degree C, dry (<8%RH) (c) 90 degree C, 80 %RH (d) 80 degree C, 80 %RH

The most important results is that, disks using Pd added Te-TeO2 (TeOx) active layer, high temperature environment of 100 degree C and also high temperature and high humidity environment of 90 degree C, 80 %RH, have no apparent defect increase, compared to other Te Te alloy compounds thus reported (6),(7).

Archival C.N.R. exhibits almost no change as is shown in Fig.5, and defects are also almost unchanged as is shown in Fig.6, then it is reasonable to say that no apparent archival

B.E.R. change is observed in Fig.6.

Figs.7(a),(b) and (c) (d) show that writable raw B.E.R. (Shelf) variation data. ditions are (a) and (b) and high humidity 80 %RH conditions are (c) and (d), respectively.

In dry conditions, at 100 degree C, after a test time of around 4 000 hours, B.E.R. (Shelf) seems to degrade slowly, and at 90 degree C, after more than 10,000 hours. In high humidity (80%RH) conditions, Shelf B.E.R. degradation is obviously observed. At 90 degree C, 80 %RH, around 800 hours, writable (Shelf) B.E.R. seems to start to degrade and, at 80 degree C 80 %RH around 2,000 hours, writable (Shelf) B.E.R. starts to degrade. It is important that these Shelf B.E.R. degradations do not correspond to defect varia-

tions as reported using Te compounds (7).

As it is shown in Fig.6, that disks using Pd added Te-TeO2 (TeOx) active layer, at high temperature and high humidity environment, such as 100 degree C, dry and 90 degree C, 80 %RH, respectively, generated no apparent defect increase. But variations of writable C.N.R. (Shelf) are observed in Fig.5. These changes correspond to recording signal amplitude vari-They seem to be sensitivity shift to higher recording power region. ations. And writable B.E.R. (Shelf) variation corresponds to writable signal amplitude variation.

From these acceleration test, it is concluded that, optical disks using Pd added Te-TeO2 (TeOx) active layer shows (1) Archival B.E.R. life is much longer than shelf life in these stress test conditions (2) Shelf B.E.R. life is estimated to be more than 10 years at 32

degree C, 80 %RH.

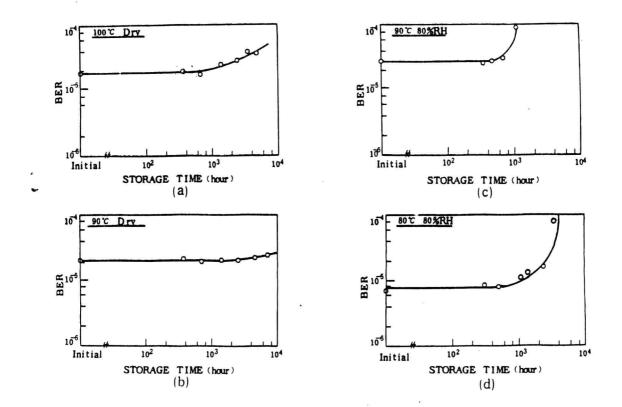


Figure 7. Acceleration test results of writable raw bit error rate (Shelf B.E.R.) as a function of storage time.

O; Shelf B.E.R.

- (a) 100 degree C, dry (<8%RH)
- (b) 90 degree C, dry (<8%RH)
- (c) 90 degree C, 80 %RH
- (d) 80 degree C, 80 %RH

5. Recording mechanism

A typical composition of the active layer was Te:50. O:35, Pd:15 (atomic %) which was analyzed by the X.P.S. measurement to be the mixture of Te, TeO2, Te, Pd, PdTe2 elements.

Fig.8 shows transition temperature of Pd added Te-TeO2 (TeOx) active layer. The film was deposited on quartz substrates and transmittance was measured by a He-Ne laser during the sample heating up to 300 degree C, by a rate of 100 degree C/min.

Fig. 8 shows that an as-deposited film had a transition temperature of around 160 to 200 degree C. By a laser beam irradiation, these thin films, absorbed the high energy and heated up above the transition temperature. Then, those optical constants n, k values changed and data could be thermally recorded.

Optical constants were determined by a multi reflection interference analysis: Obtained

refractive index, n1=3.10 and extinction coefficient k1=1.17, respectively. After a heat treatment of 250 degree C, 5 minutes, the active layer changed to darken state, and the optical constants changed to n2=3.87 and k2=1.62, respectively.

Fig. 9 shows calculated reflectivity curve vs. the film thickness. The configuration for calculation is that the substrate having refractive index of n=1.58 and above it Pd added Te-TeO2 (TeOx) active layer and above the film, air n=1.0.

Reflection changes of the active layer were estimated to go up to $\triangle R = R2 - R1 = 15$ % at a film thickness of 150 nm by thermal transition to darkening state.

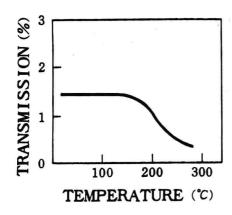


Figure 8. Transmission temperature measure- Figure 9. Calculated reflectivity variament of Pd added Te-TeO2 (TeOx) active layer. tion s as a function of the film thickness Measurement conditions 100 degree C/min. $\lambda = 633$ nm

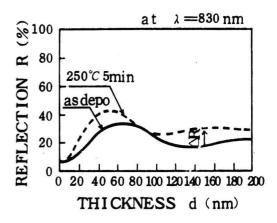
The film structure of the active layer was observed by transmission electron microscope (T.E.M.).

Fig.10(a) shows the image and diffraction pattern of the as-deposited active layer.

A uniform film structure was observed. film is formed by amorphous TeO2 network within fine PdTe2 and Te crystalline particles dispersed. As confirmed by the electron difthe inner diffraction fraction corresponds to PdTe2 fine polycrystalline (hexagonal) structure.

Fig.10(b) shows grain size growth of PdTe2 (after a heat treatment of 250 degree C 5 min.) and Laue diffraction spots are observed in the diffraction ring. It shows that the recording can be performed by the successive crystalline grain growth of PdTe2 without amorphous TeO2 network change.

As mentioned before, by T.E.M. observation, the recorded dots show some crystalline grain growth and crystalline diffraction spots appear in the diffraction ring. It is coincident with the diffraction pattern of the film which thermal transition have occurred.



at $\lambda = 830$ nm.

 as deposited after annealing at 250 degree C 5min.

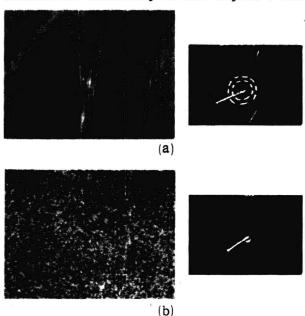


Figure 10. Transmission electron micrographs and diffraction patterns of Pd added Te-TeO2 (TeOx) active layer.

(a) as deposited film

(b) after annealing at 25J degree C 5min.

outical disk using Pd added Te-TeO2 (TeOx) active layer shows a long life by the activition stress tests. Here about active layer, more severe stress test was carried out. he active layers were deposited on quartz and polycarbonate substrates, and put into is summent test chamber without any protection.

The test conditions are 110 degree C dry (<8 %RH) 100 hours, (b) 80 degree C, dry (<8 % RH) 300 hours and 80 degree C, 80 %RH, 300 hours, respectively.

transmittance and transition temperature of this films were measured for the samples so the quartz substrates.

fig.11(a) shows 110 degree C, dry, 100 hours stressed sample data, (b) 80 degree C. dry

(<8 %RH) 300 hours stressed sample data and (c) for 80 degree C 80 %RH, 300 hours stressed sample data.

At 110 degree C, dry testing transmission degrades comparably large and the transition temperature looks like to shift to a higher temperature, and at 80 degree C, dry, testing, the degradations are small and at 80 degree C. 80 %RH smaller than that of 80 degree C,

The transmission slightly degrades and also the transition temperature looks like shift

up to 190 degree C.

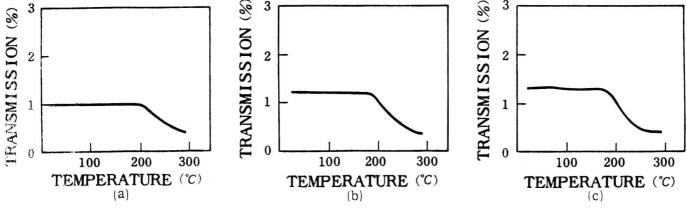


Figure 11. Transition temperature measurement of Pd added Te-TeO2 (TeOx) active layer after acceleration tests.

Measurement conditions; 100 degree C/min., λ =633 nm

(a) 110 degree C, dry (<8%RH)

300 hours

(b) 80 degree C dry (<8%RH) (c) 80 degree C, 80 %RH 300 hours

It is concluded that the writing sensitivi -ty degradation mechanism of Pd added Te-TeO2 (TeOx) active layer corresponds to this transition temperature shift to a higher temperature. The other samples deposited on the polycarbonate substrates were observed by a T.E.M. (transmission electron microscope).

Fig. 12(a) shows the film structure of sample tested at 110 degree C, 100 hours. A small amount of crystalline grains are observed. Fig.12(b) shows the film structure of the sample tested 80 degree C, 80 %RH, 300 hours, and no noticeable structure change are observed. Both samples show no defect generation originated from the oxidized corrosion in the film.

It is also concluded that the thermal degradation in the dry and the high humidity environments, is originated from the some amounts of grain growths of PdTe2 crystallines, and it corresponds to recording sensitivity changes, and no corrosion defect increase corresponds to no defect distribution change of the disks.

In high humidity environmental tests uniform increase of oxigen is expected, and this phenomena may cause a sensitivity degradation also.



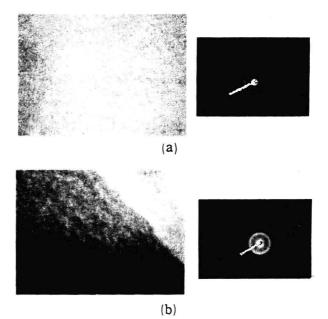


Figure 12. Transmission electron micrographs and diffraction patterns of Pd added Te-TeC2 (TeOx) active layers after acceleration tests.

(a) 110 degree C dry (<8%RH) 100 hours (b) 80 degree C 80 %RH , 300 hours

7. Discussions

The model of rapid response of Pd added Te-Teo2 (TeOx) active layer is considered to be that solidification occures in short time because the film contains high melting temperature elements.

Those high melting temperature components are PdTe (Tm=720 degree C) and PdTe2 (Tm=740 degree C), respectively. After pulsed laser irradiation, the film is heated and the center region of the recorded dots melts and cooling starts successively to under the melting temperature. They change to solid state in short time and the grain growth finish and the transient phenomena will be eliminated.

On the other hand. Te-TeO2 (TeOx) active layer as the melting temperature of $\,$ Te $\,$ is $\,$ 449 degree C, therefore in the cooling process, Te crystalline grain growth occures and successively re-orientation of Te grains occurs in the film and gradually amplitude increasing phenomena appears.

The bit error rate level of the disks used in this experiment is within the range of 1 $\,\mathrm{X}$ E-6 to 1 X E-4, comparably high B.E.R. level, but it is not intrinsic characteristics, well arranged disk process raw B.E.R. level is within the range of 1 X E-6 is accomplished.

The Shelf B.E.R. degradation mechanism is understood by shelf amplitude degradations

caused from sensitivity change.

As reported before Te-TeO2 (TeOx) active layer, the transition temperature goes up at the value increasing (4) so in high temperature and 80 %RH, x value increase and both thermal

and x value changes are considered to affect the sensitivity shift.

8. Conclusions

Optical disks using sub-oxide Te-TeO2 (TeOx) containing Pd additive active laver shows high speed response (300 n sec) characteristics.

From the disk environmental stress test data we conclude that more than 10 years archival and shelf B.E.R. life can safely be extrapolated to at 32 degree C, 80 %RH storage condition.

The recording mechanism is that PdTe2 crystalline grain growth in amorphous Teo2 netk. The degradation mechanism is that, sensitivity change. The sensitivity shift is work. caused by thermal change of PdTe2 and also by oxidization.

Stable B.E.R. characteristics is caused from stable archival C.N.R. characteristics and no defect variation.

The Pd added TeOx active layer is expected to be a reliable optical disk which has low B.E.R. and long life.

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