

# 放射光時間分解X線回折測定による 光記録媒体の相変化構造計測

田中 義人

独立行政法人理化学研究所・播磨研究所 放射光科学総合研究センター  
JST-CREST

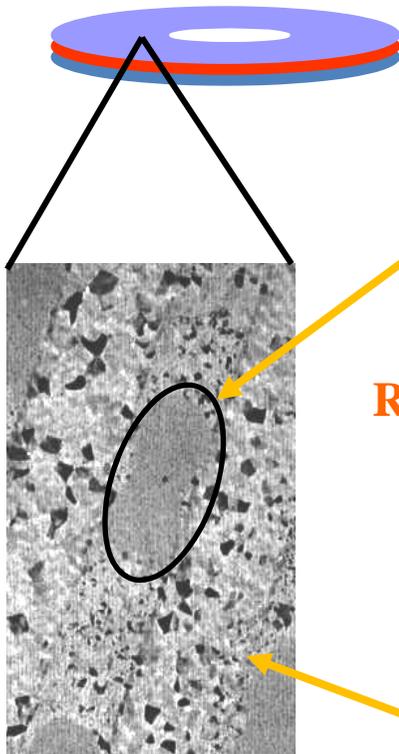
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- 1. はじめに
    - ・DVD材料における高速相変化
    - ・XRDで構造変化をみる
  - 2. 時間分解測定法の開発
    - ・放射光の時間構造と手法
    - ・時間分解X線回折測定技術
  - 3. 観察結果
    - ・反射率変化と相変化の関係
    - ・GST, AISTにおける結晶成長の相違
  - 4. まとめ
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# 1. はじめに

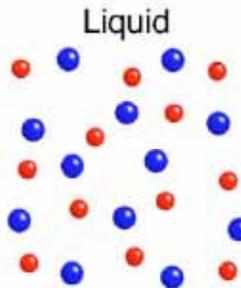
## 1.1 DVD材料における高速相変化

GST225 ( $\text{Ge}_2\text{Sb}_2\text{Te}_5$ )

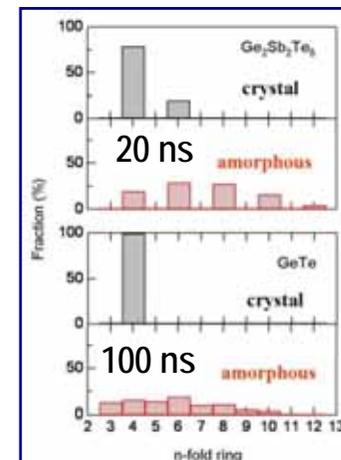
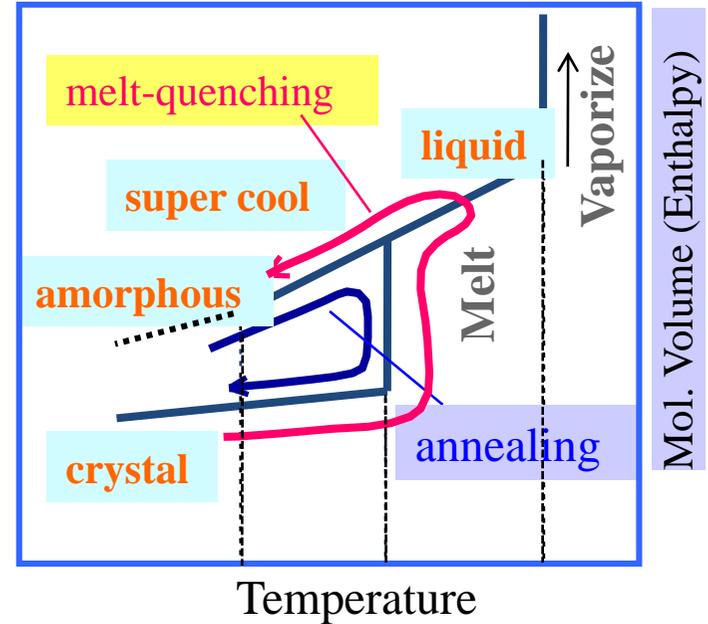
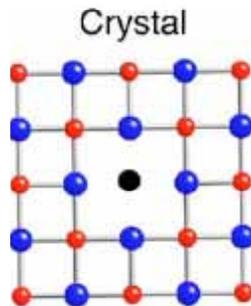
AIST ( $\text{Ag}_{3.5}\text{In}_{3.8}\text{Sb}_{75.0}\text{Te}_{17.7}$  (%))



Writing



Recorded mark  
20-30 ns Erasing

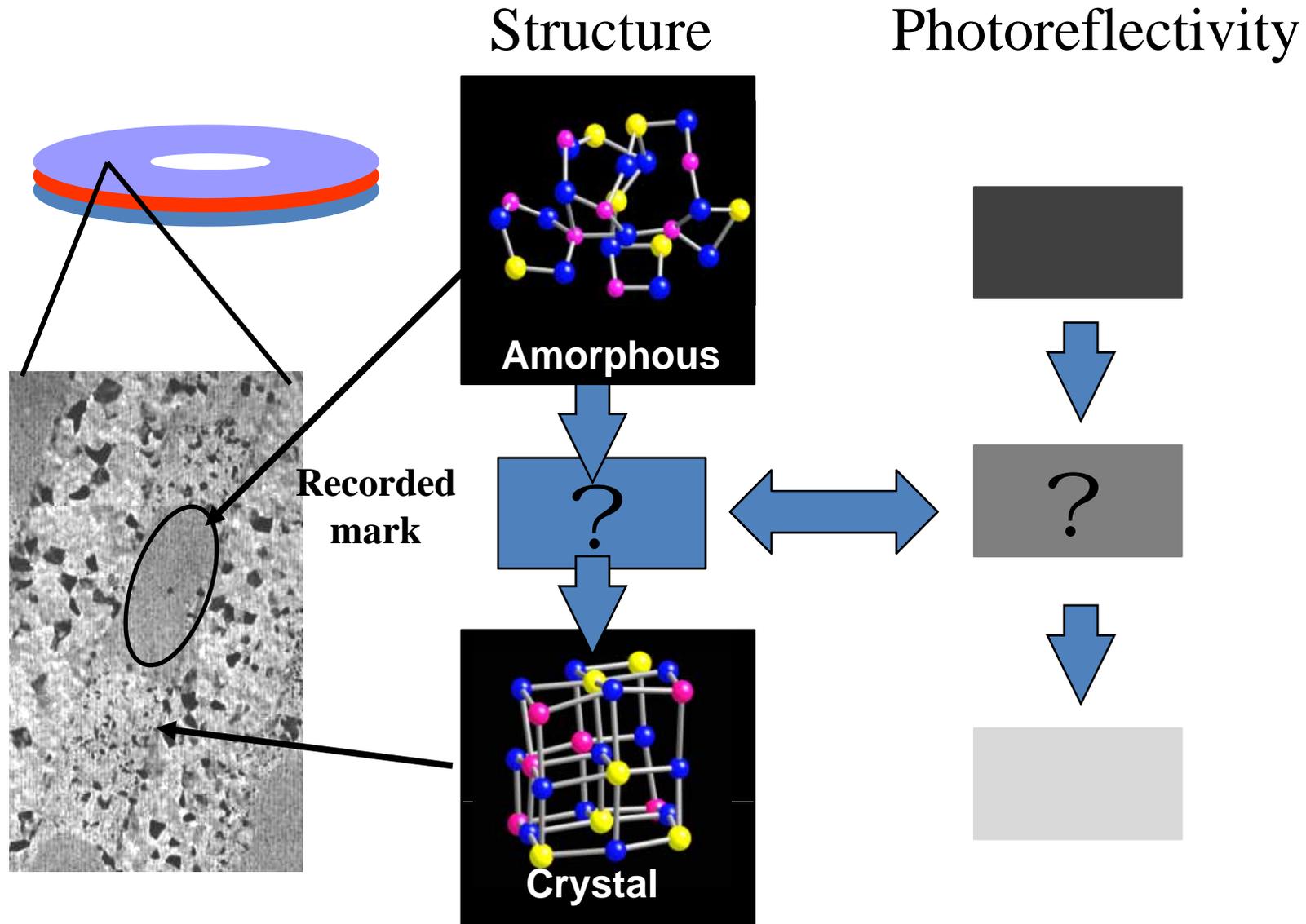


S. Kohara et al. : Appl. Phys. Lett. 89, 201910 (2006)

記録マークのサイズ:  $0.32 \mu\text{m}$   
書込・消去の速度: 20 ~ 30 ns

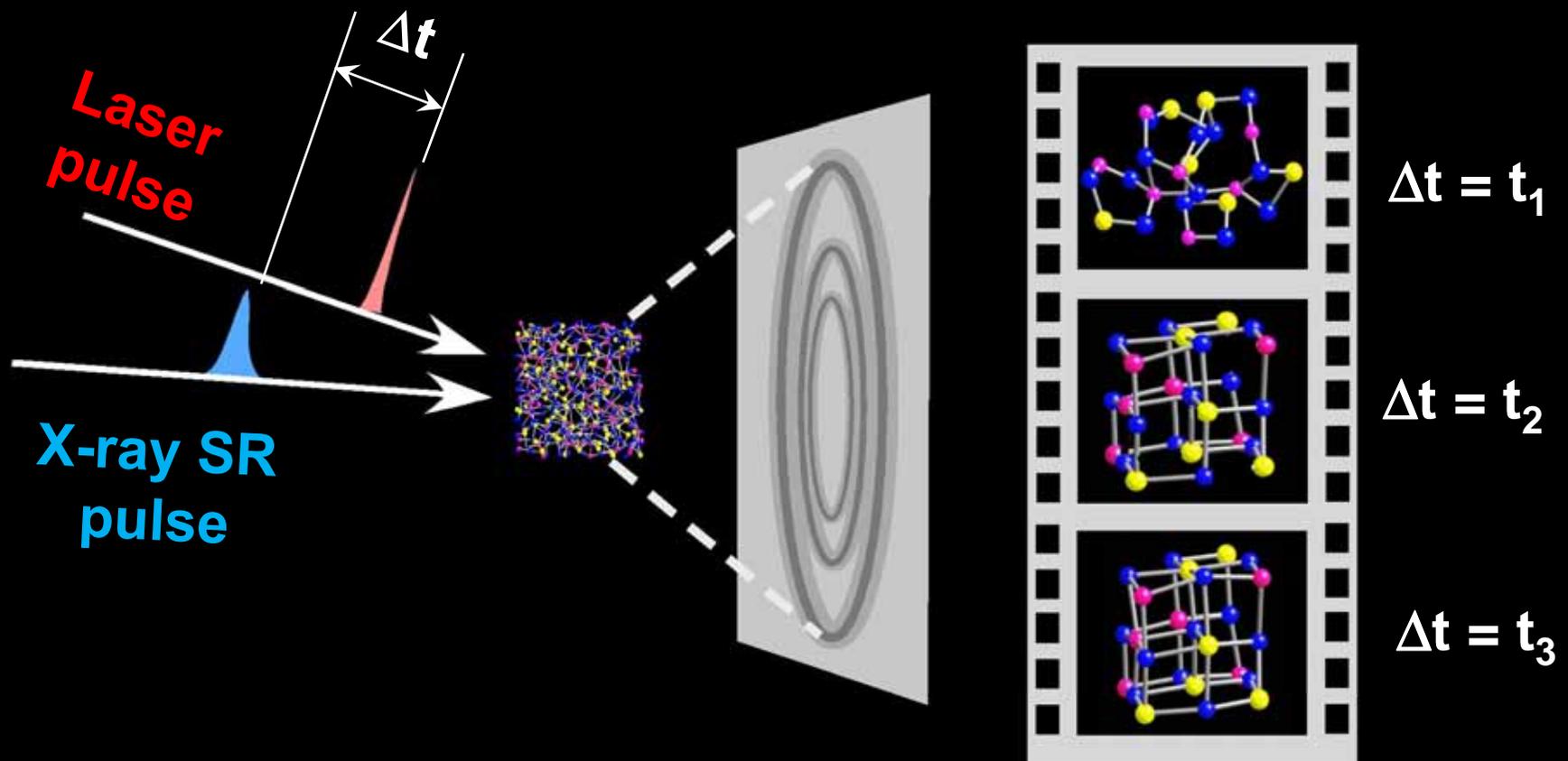
相変化速度とアモルファスのリング構造の相関

# 反射率と構造の関係？



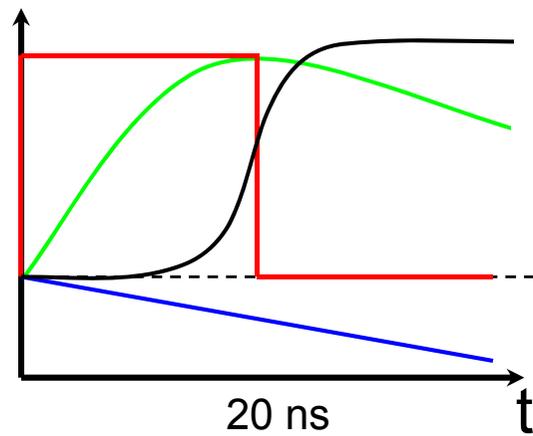
## 1.2 XRDで構造変化をみる

### Time-resolved X-ray diffraction (Laser pump-X-ray SR probe)

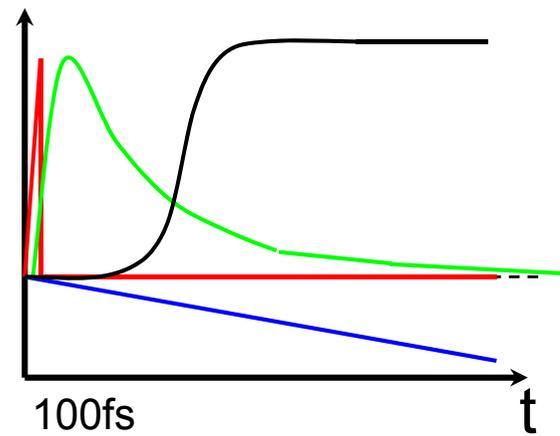


# インパルス応答を調べる

## Commercial DVD media



## Impulse response



- Laser irradiation
- Thermal diffusion
- Temperature
- Diffraction Intensity

**フェムト秒パルスレーザー  
による刺激**

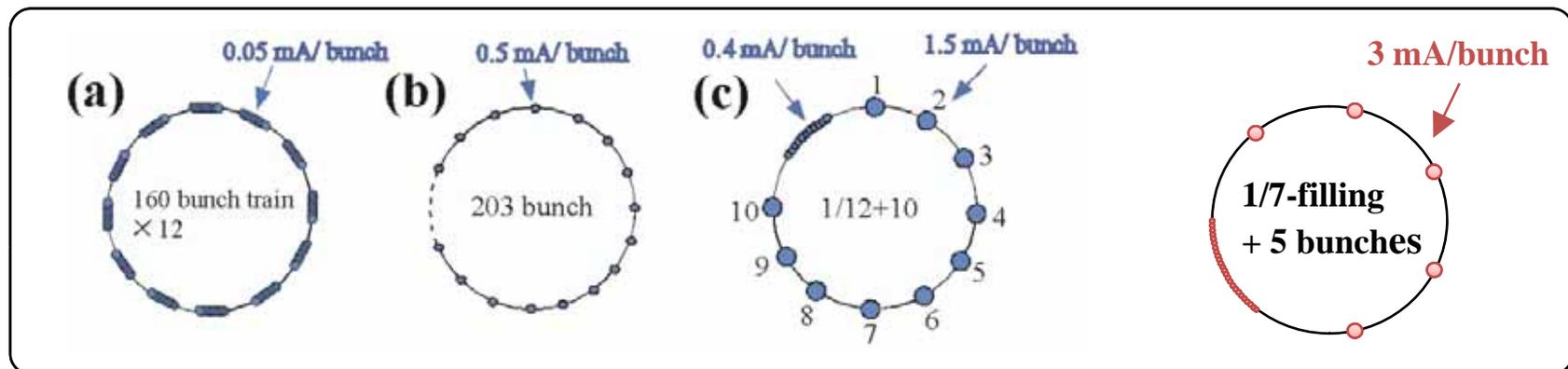
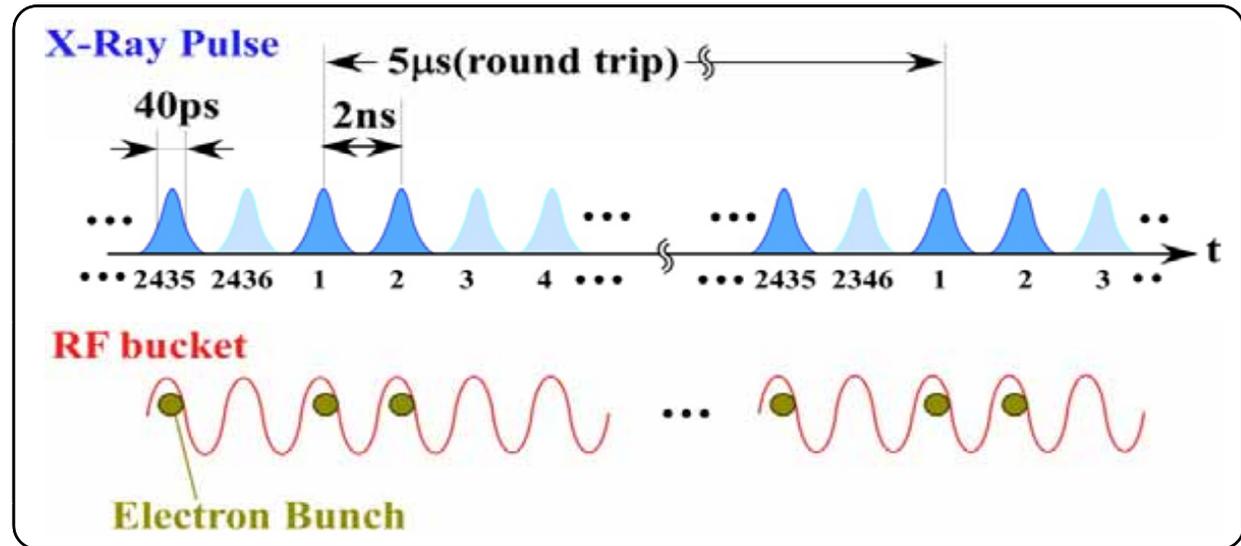
## 2. 時間分解測定法の開発

### 2.1. 放射光の時間構造と手法

#### 蓄積リング放射光の時間構造



**Pulse width:**  
40 ps (FWHM)  
**Rep. rate:**  
200 kHz-509MHz

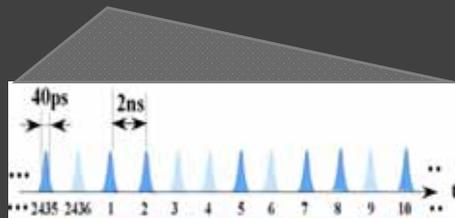


# 2.2. 時間分解X線回折測定技術

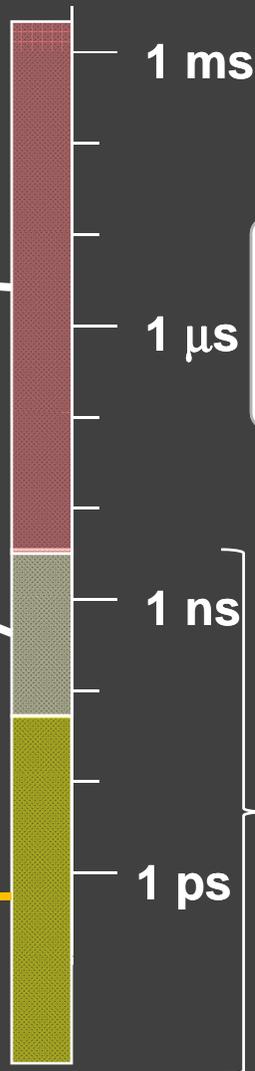
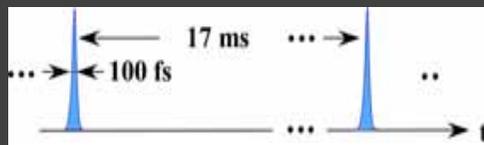
## SR の時間構造

## 時間分解能と手法

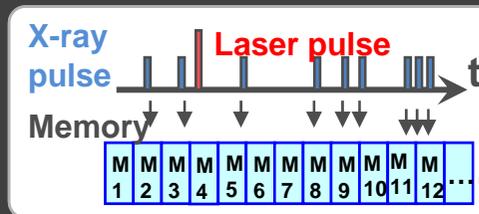
### Storage Ring



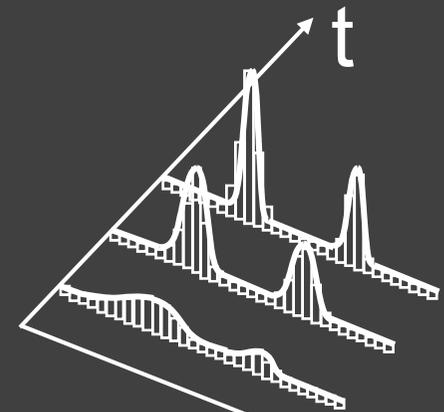
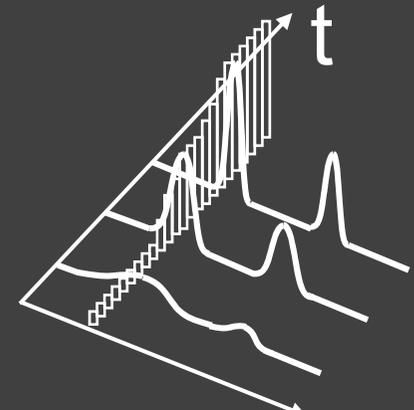
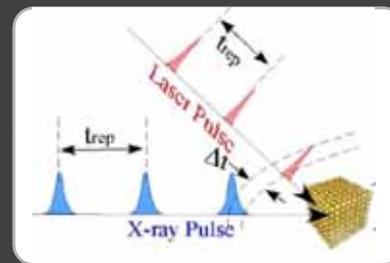
### XFEL



### APD+MCS



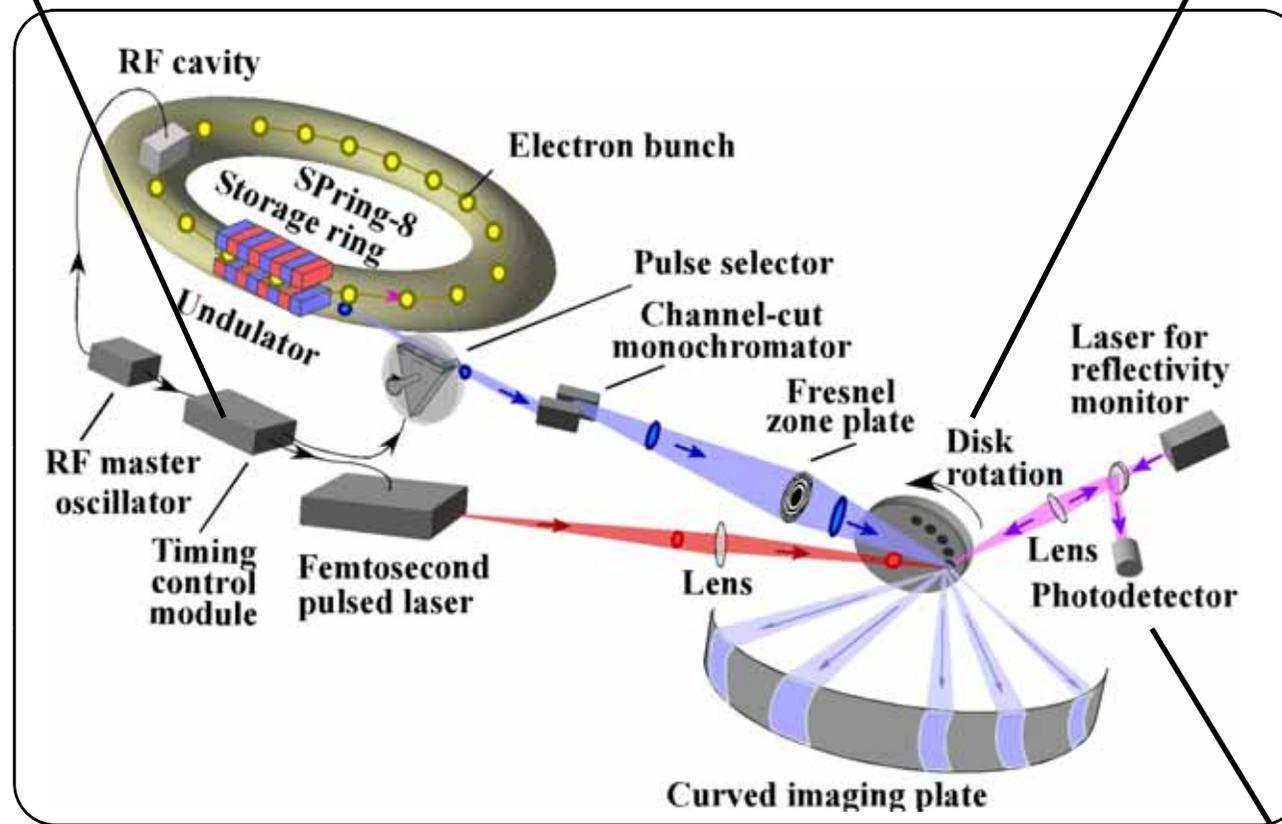
### Pump-probe



# 要素技術

(1) Timing control between SR and laser pulses

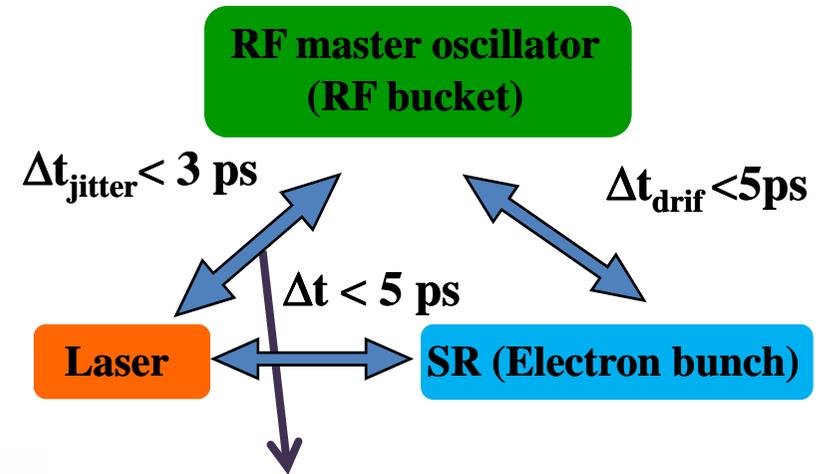
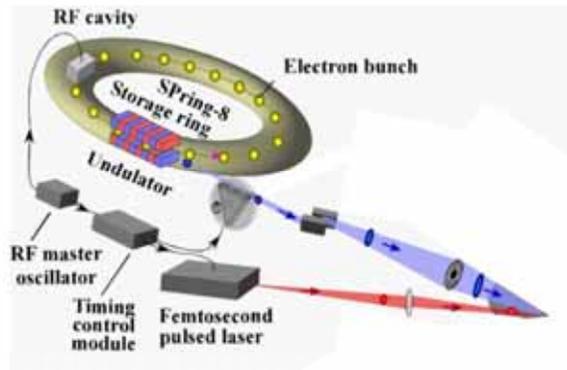
(2) Sample rotation stage for phase-change materials



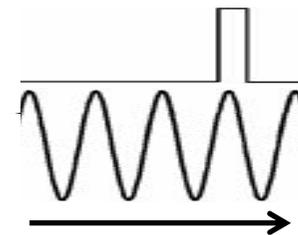
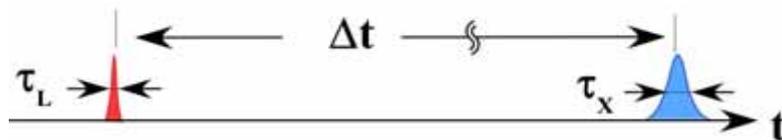
(3) On-line monitor of photorefectivity

# (1) Timing control between SR and laser pulses

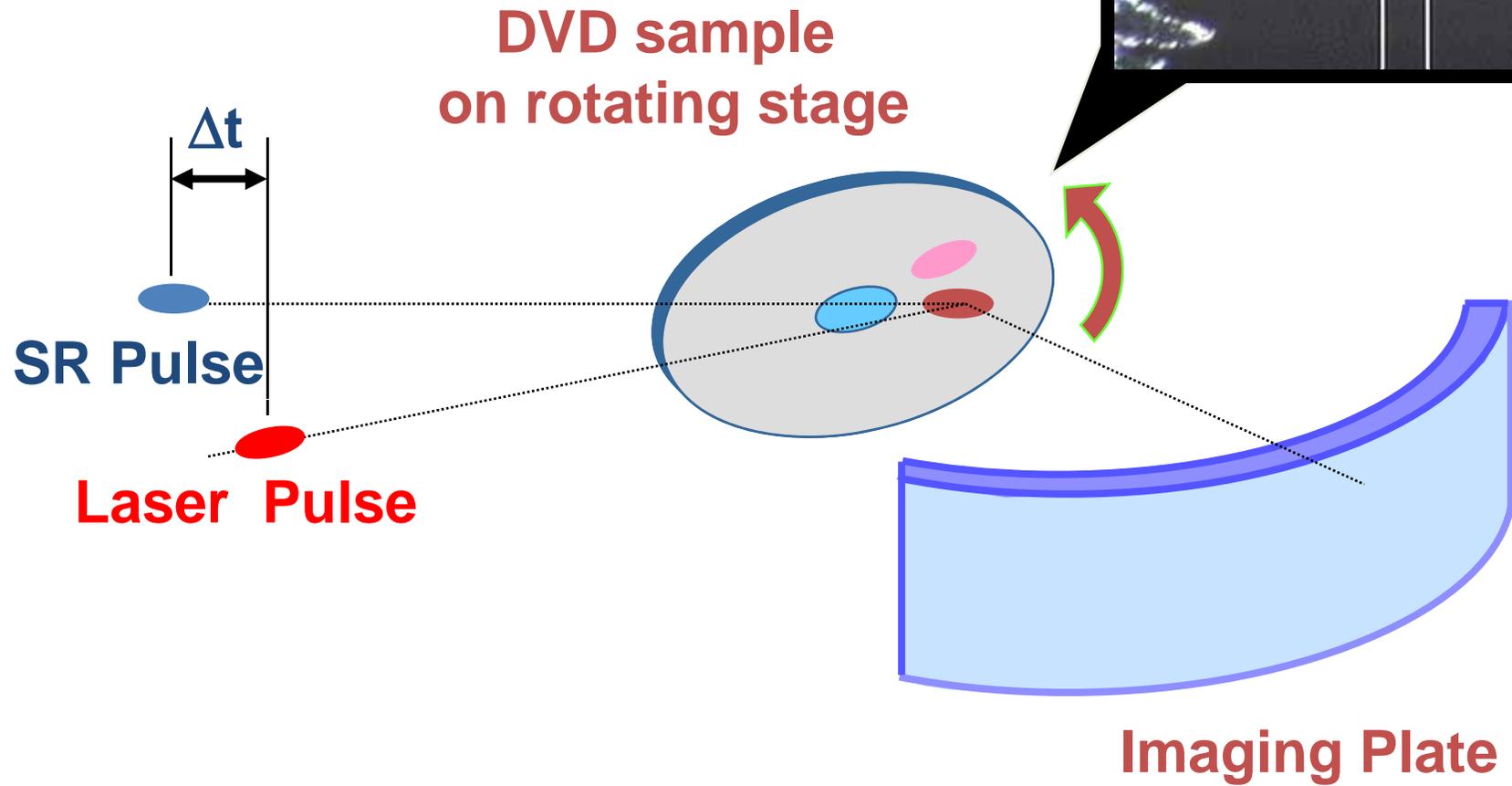
## (i) Synchronization of laser and SR



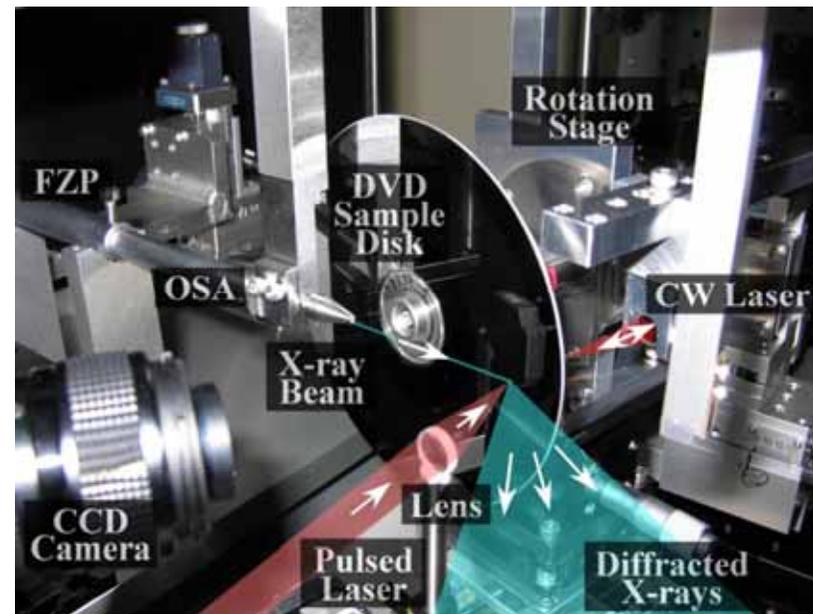
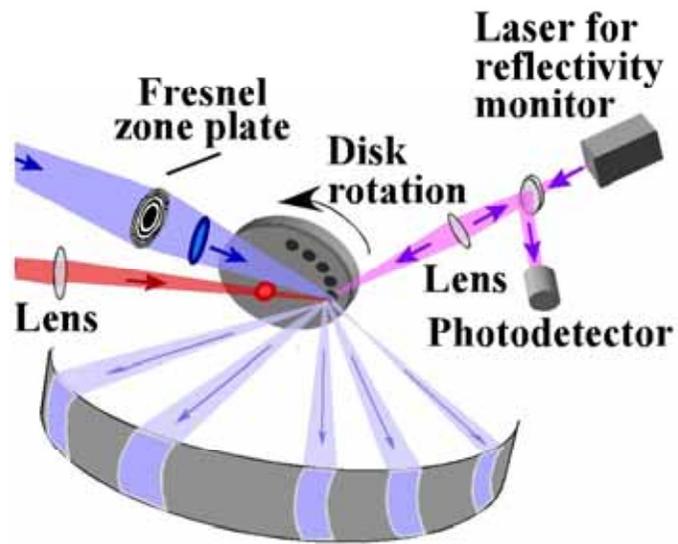
## (ii) Time delay control



**(2) Sample rotating system**



### (3) On-line monitor of photorefectivity

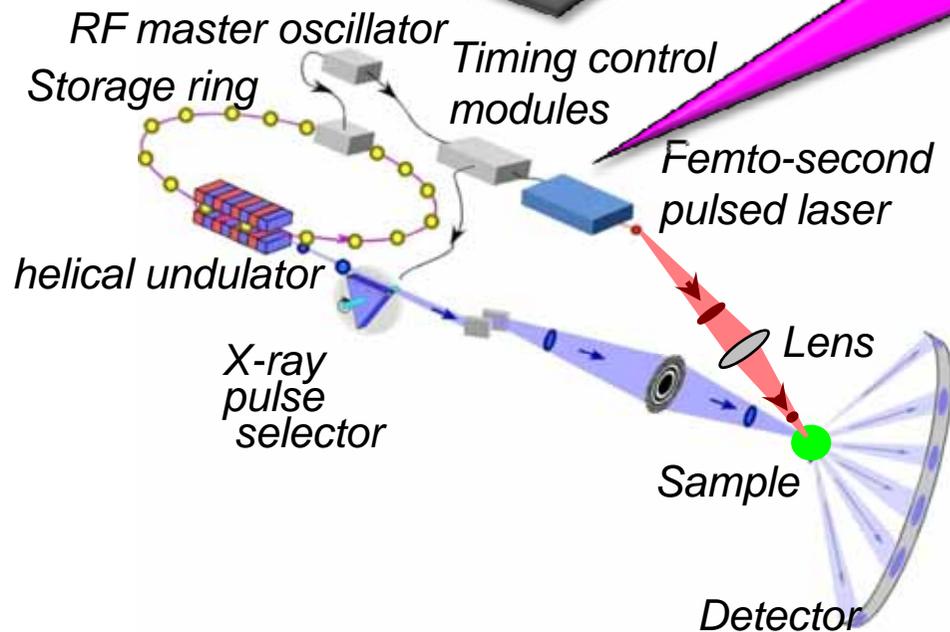


# Picosecond TR-XRD system

Timing control circuit



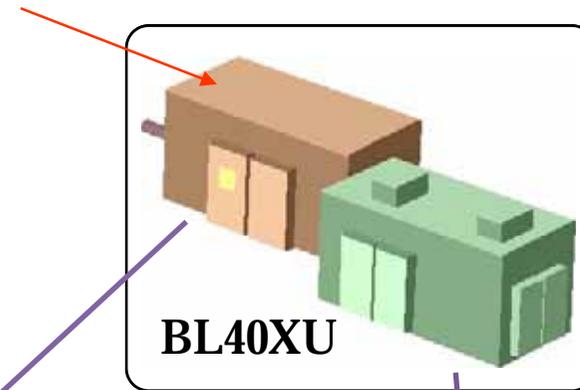
Femto- pico-second laser system



Diffractometer



# Time-resolved diffraction station in BL40XU

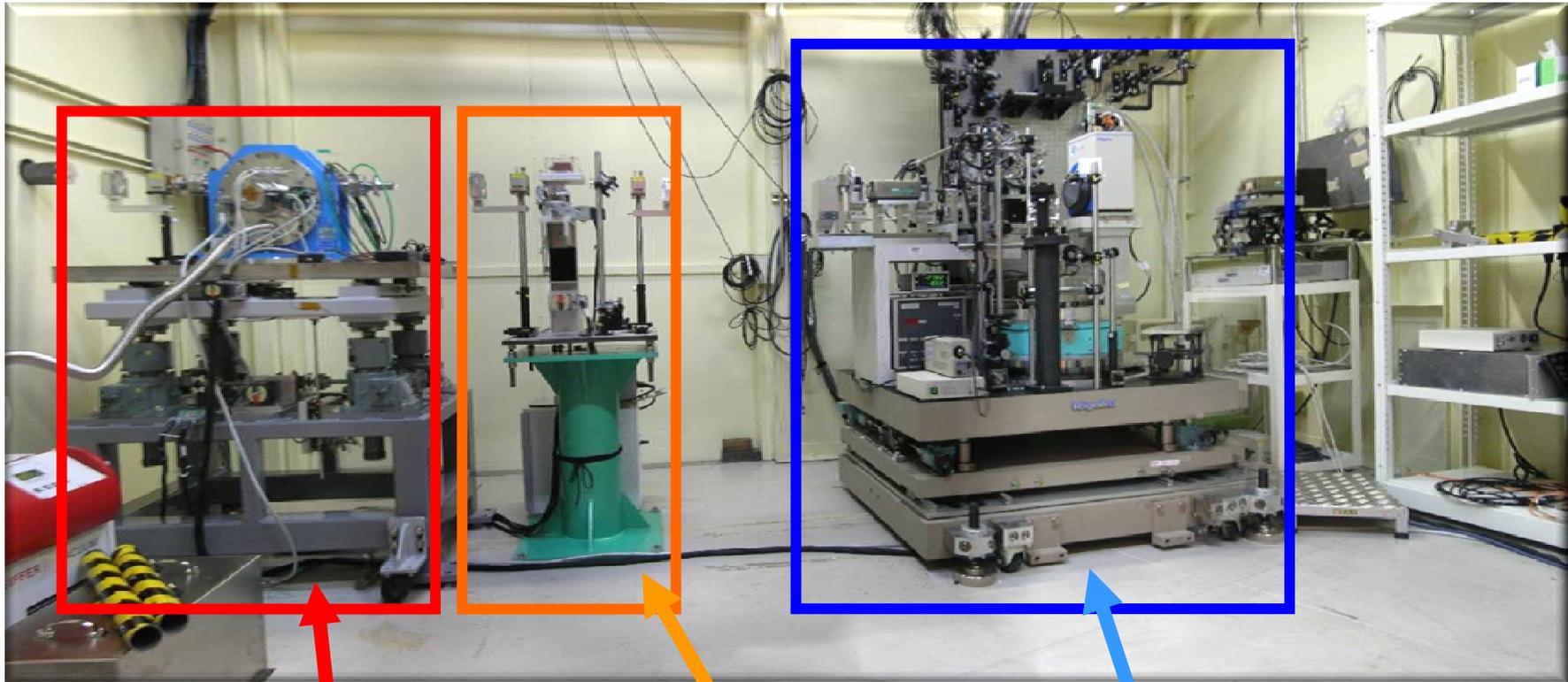


**X-ray exp. hutch**



**Laser booth**

## X-ray experimental hutch@ SPring-8 BL40XU

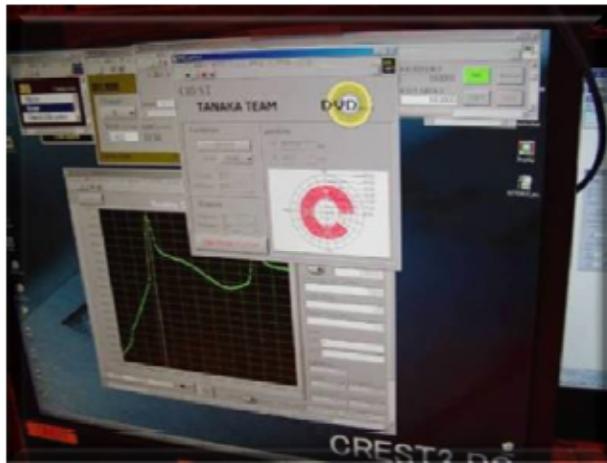


X-ray Pulse Selector (XPS)

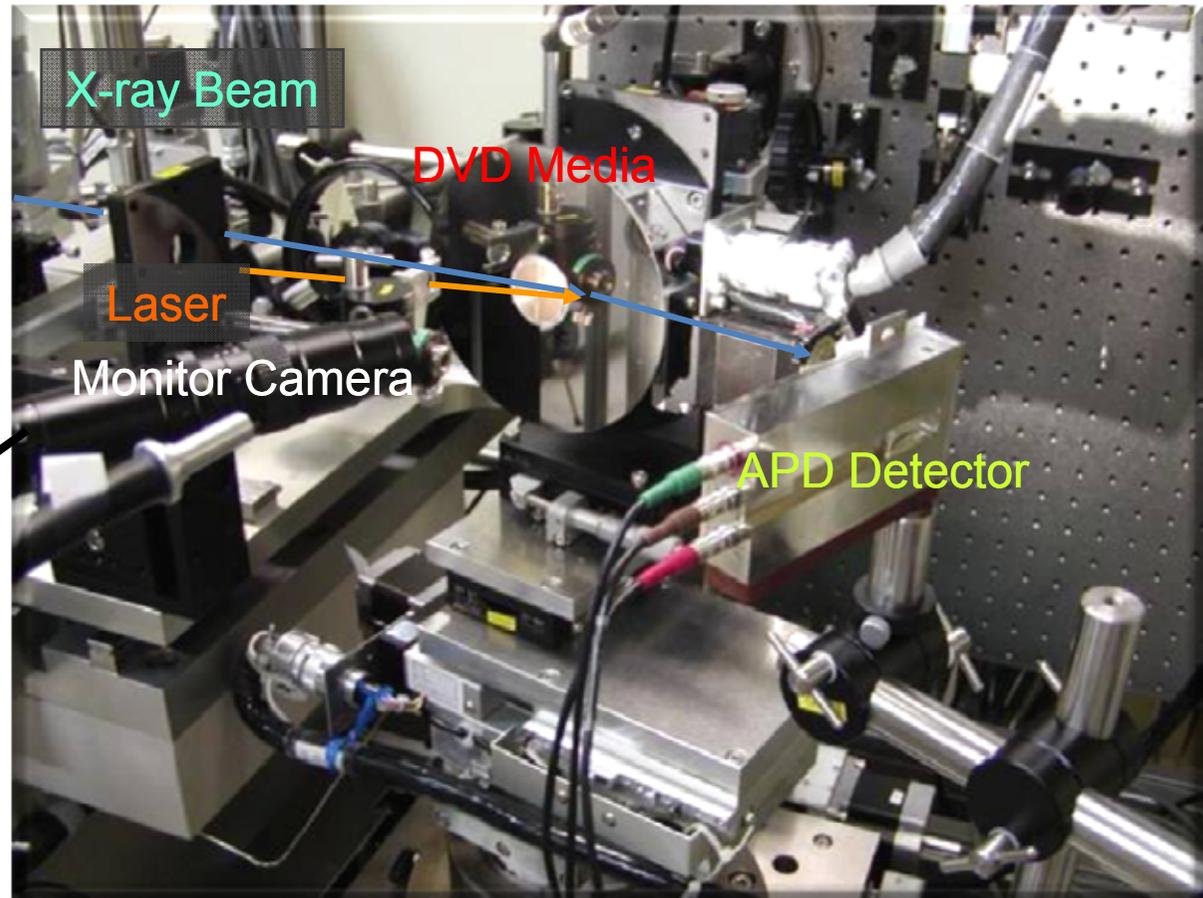
Si(111) Channel-cut Monochromator

Precise Diffractometer System

# DVD Phase Change Time Resolved X-ray Measurement System

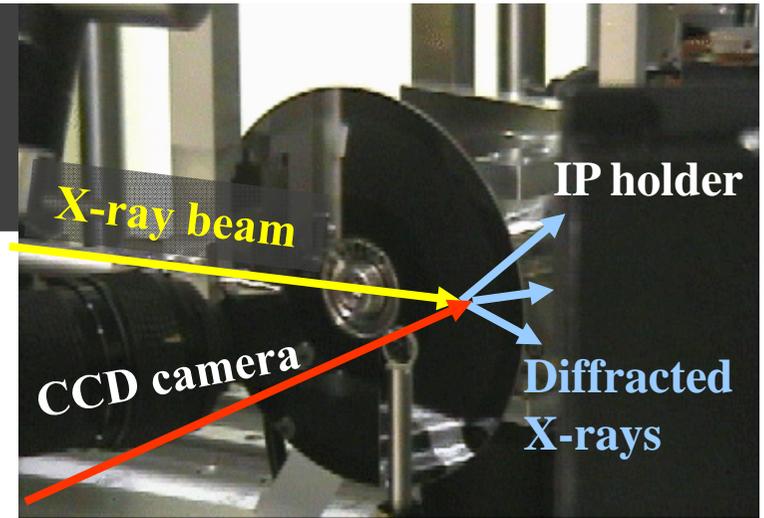


**Single Shot Controller**

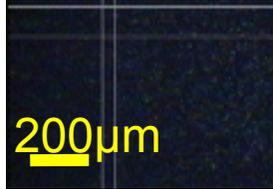


**Sample Monitor**

# DVD材料測定用高速回転試料台



**Nov. 2006**  
**Repetition rate 1 Hz**



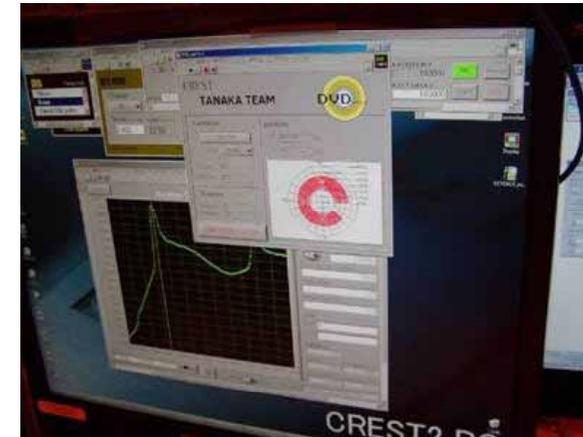
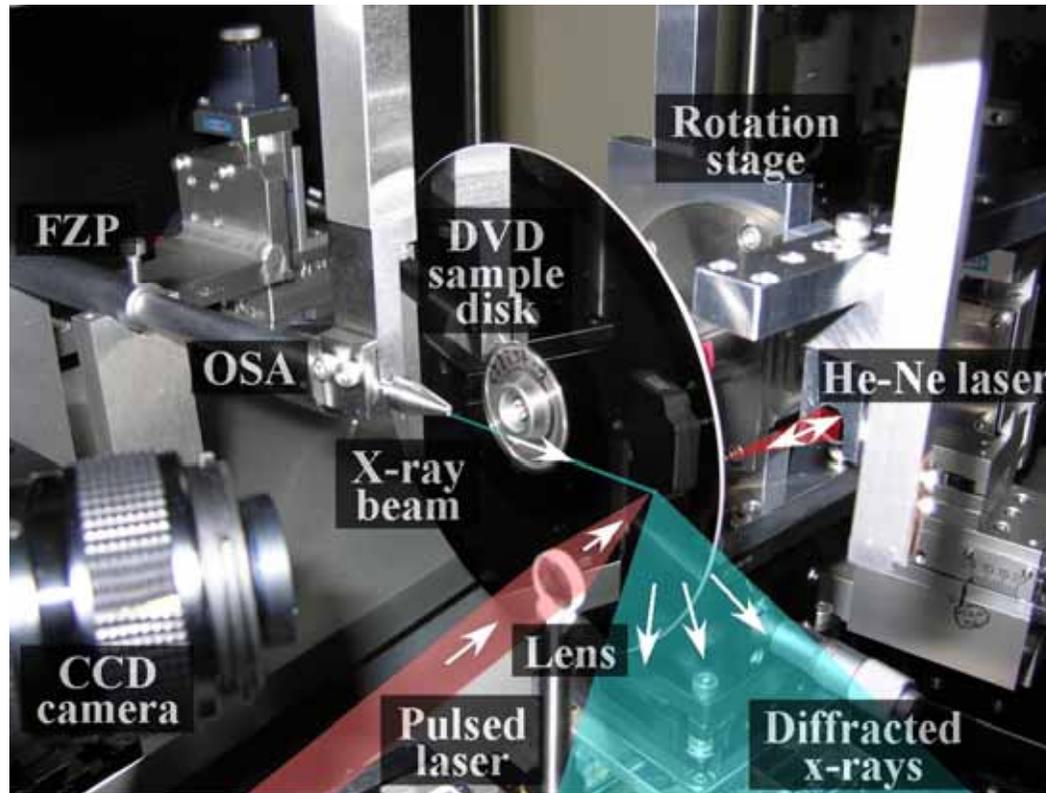
**April 2007**  
**Repetition rate 5 Hz**



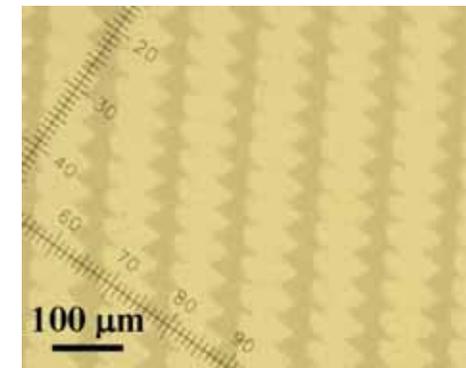
**April 2008**  
**Repetition rate 1 kHz**  
**Using X-ray microbeam**



# DVD相変化材料測定用 時分割マイクロビームX線回折システム



LabVIEWによる制御画面

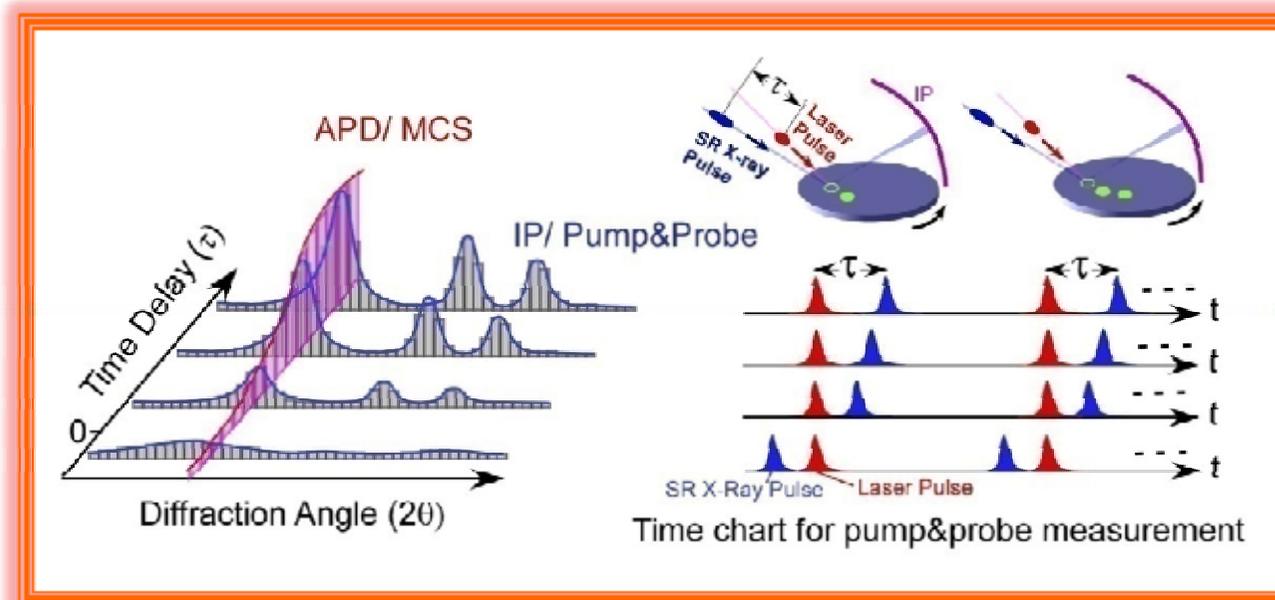
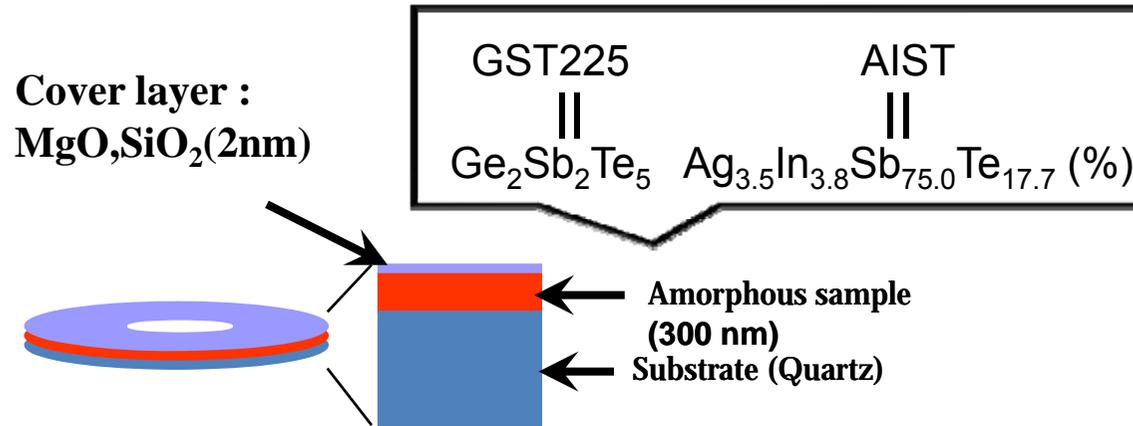


試料表面

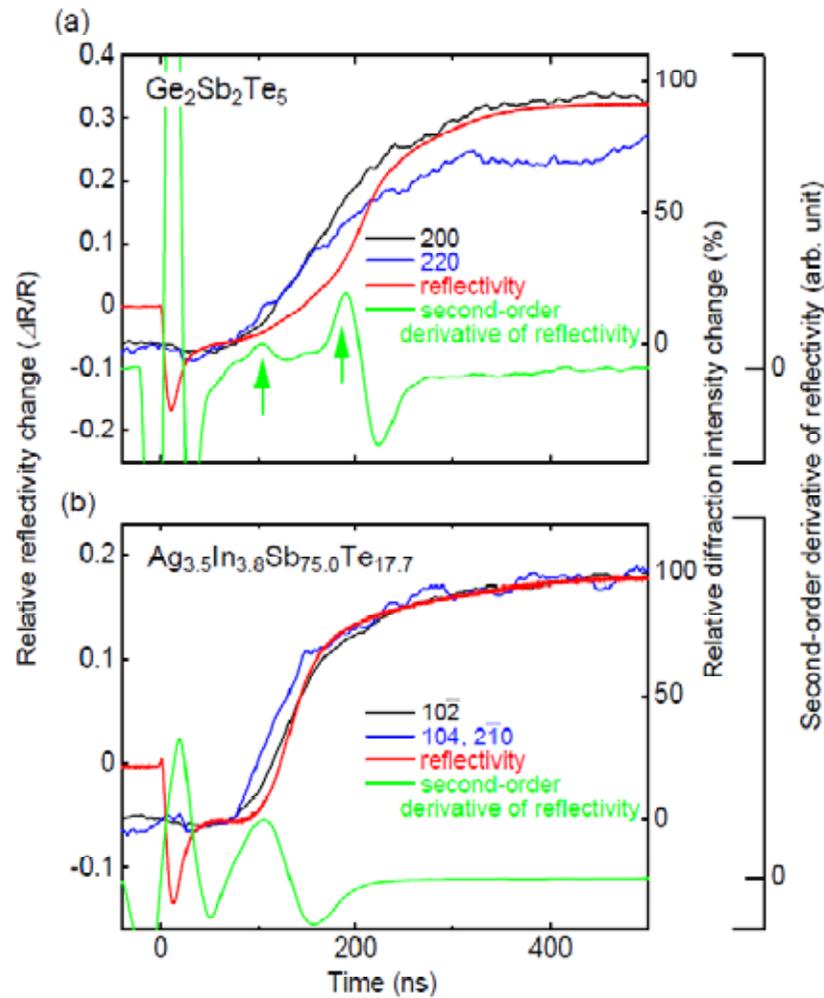
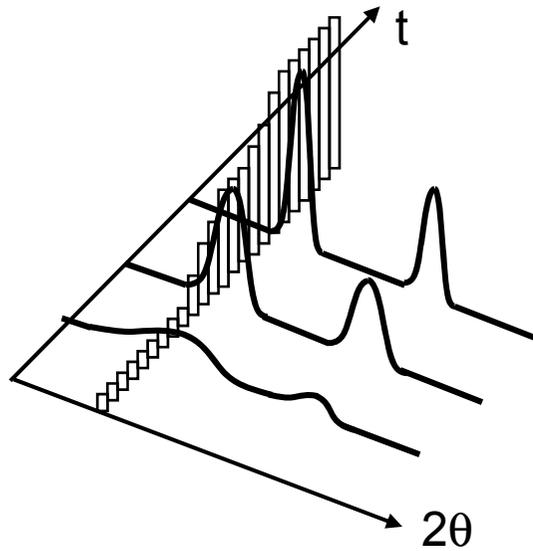
繰り返し周期 $t_{rep}$	移動速度 $v$	レーザー スポット径 $a$	X線 スポット径 $b$	ショット数 (DVD一枚あたり)	測定時間 (DVD一枚あたり)
1 Hz	1 mm/s	300 $\mu\text{m}$	100 $\mu\text{m}$	30000	10時間
5 Hz	5 mm/s	300 $\mu\text{m}$	100 $\mu\text{m}$	30000	1時間30分
1 kHz	50 mm/s	30 $\mu\text{m}$	3 $\mu\text{m}$	1800000	30分

# 3. 観察結果

## 3.1 反射率変化と相変化の関係



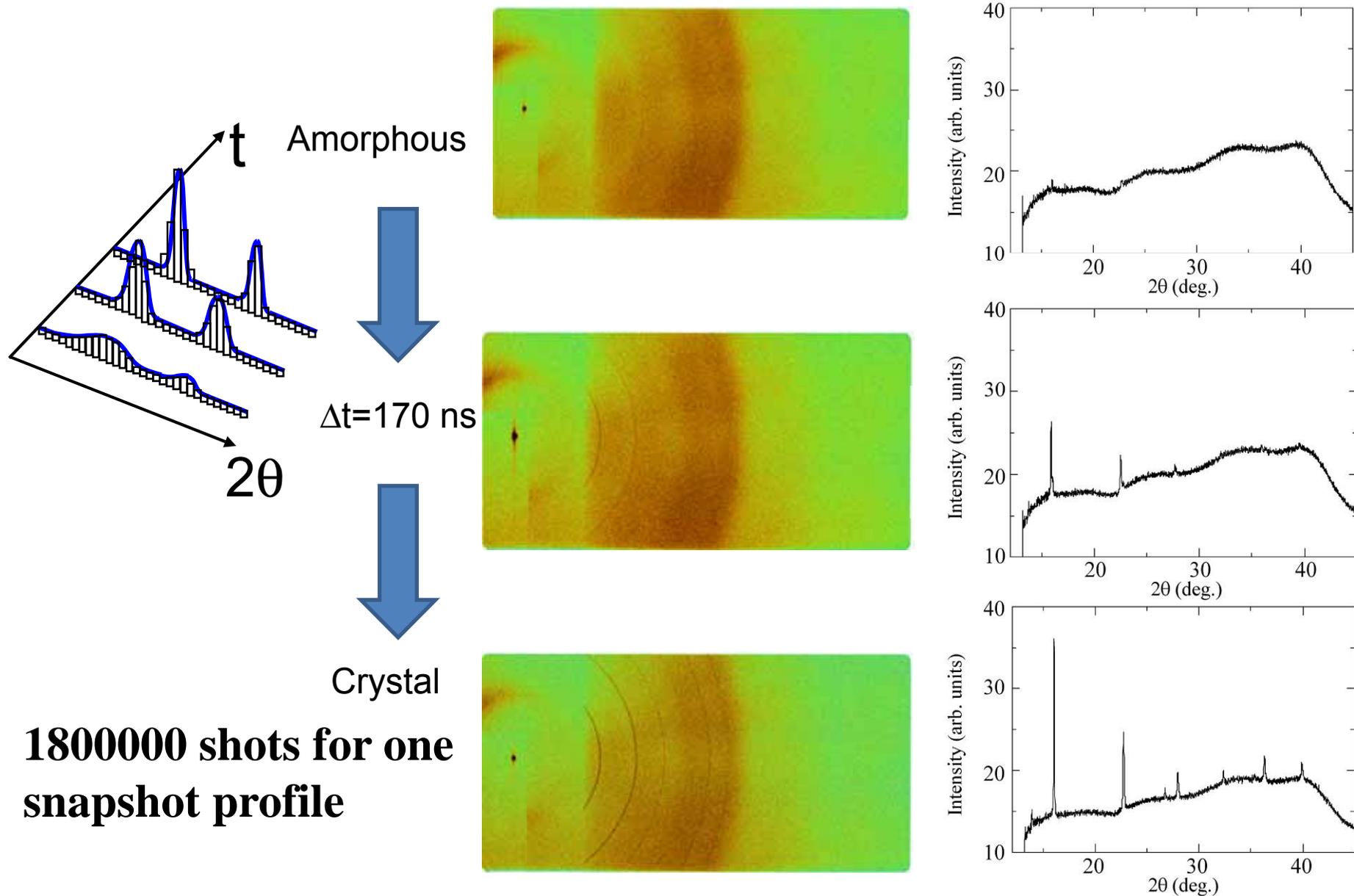
# Time dependence of diffraction intensity and reflectivity



開始 : 90 ns  
終了 : 273 ns

開始 : 85 ns  
終了 : 206 ns

# Snapshots of X-ray diffraction patterns for GST225



# Snapshots of X-ray diffraction patterns for AIST

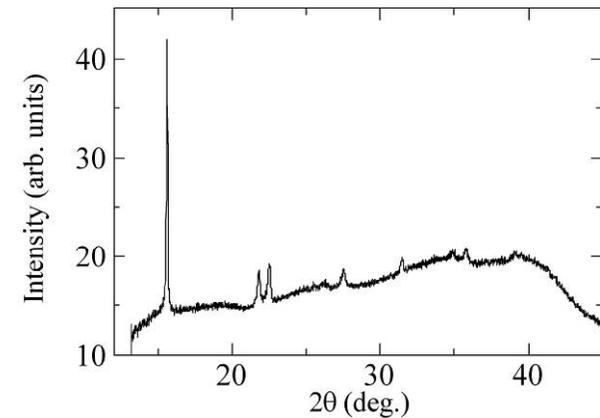
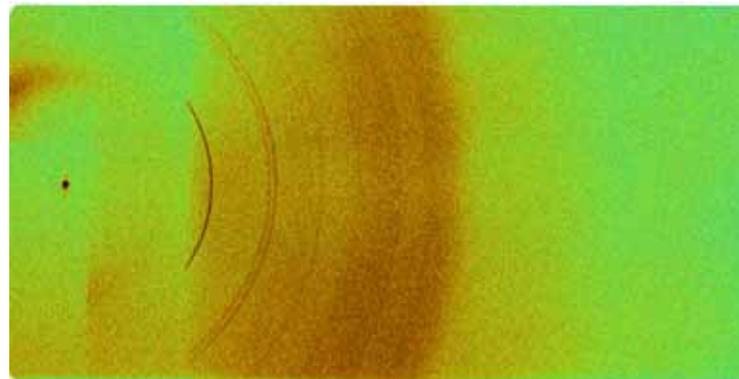
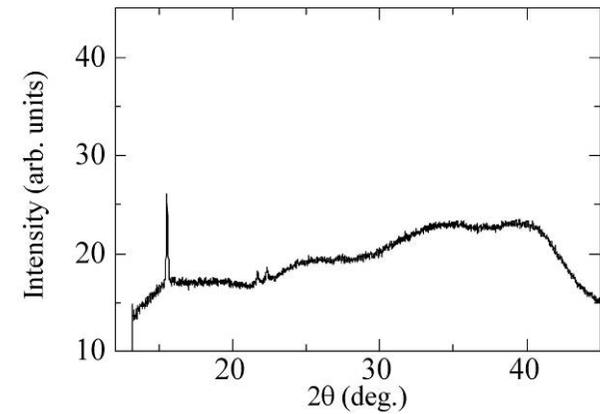
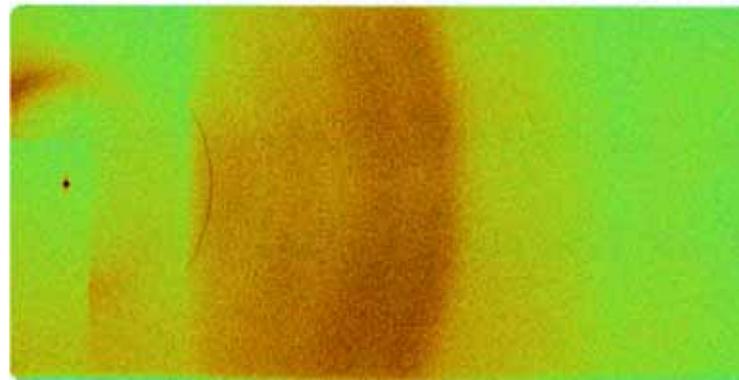
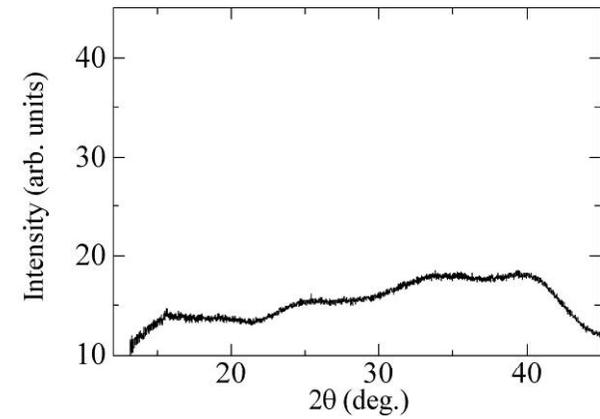
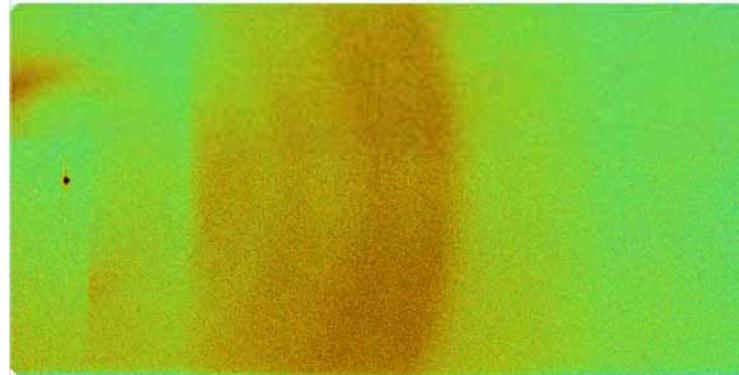
Amorphous



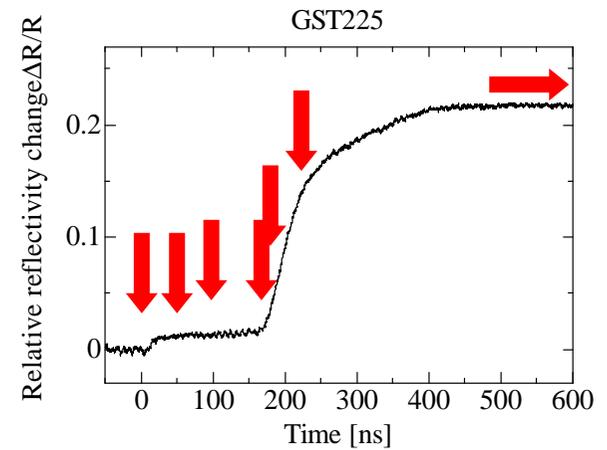
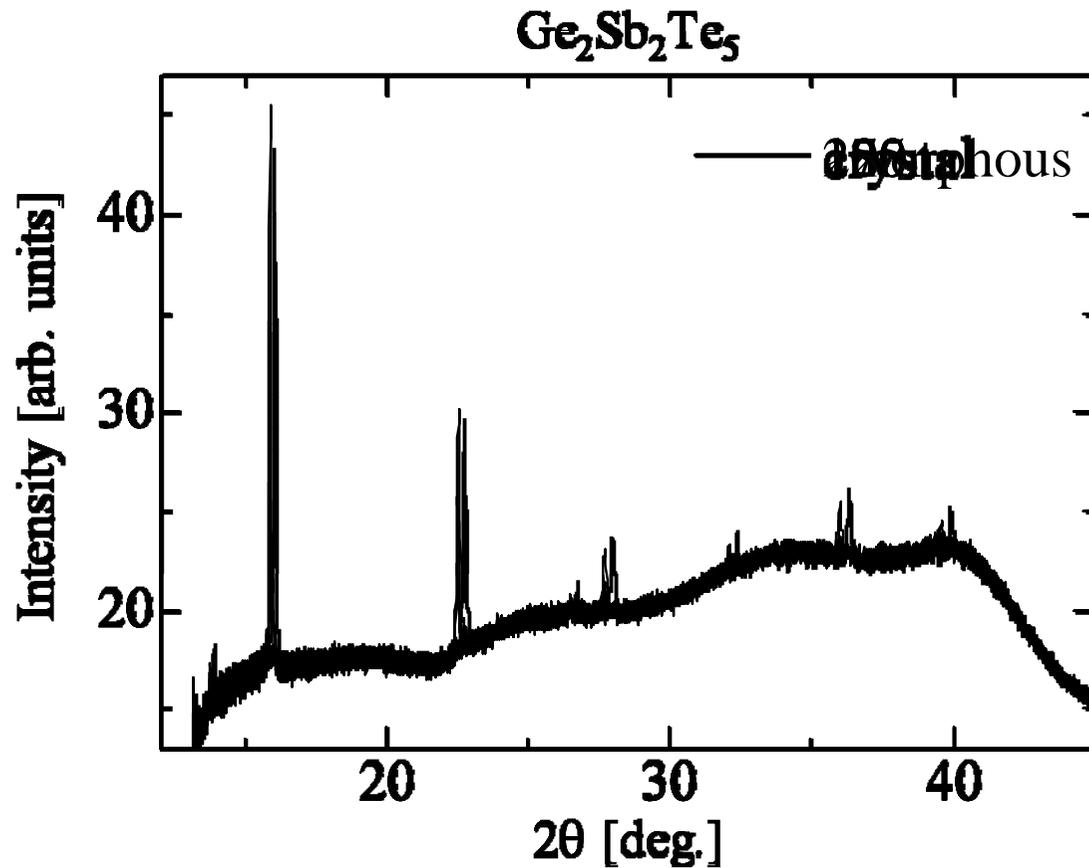
$\Delta t = 115$  ns



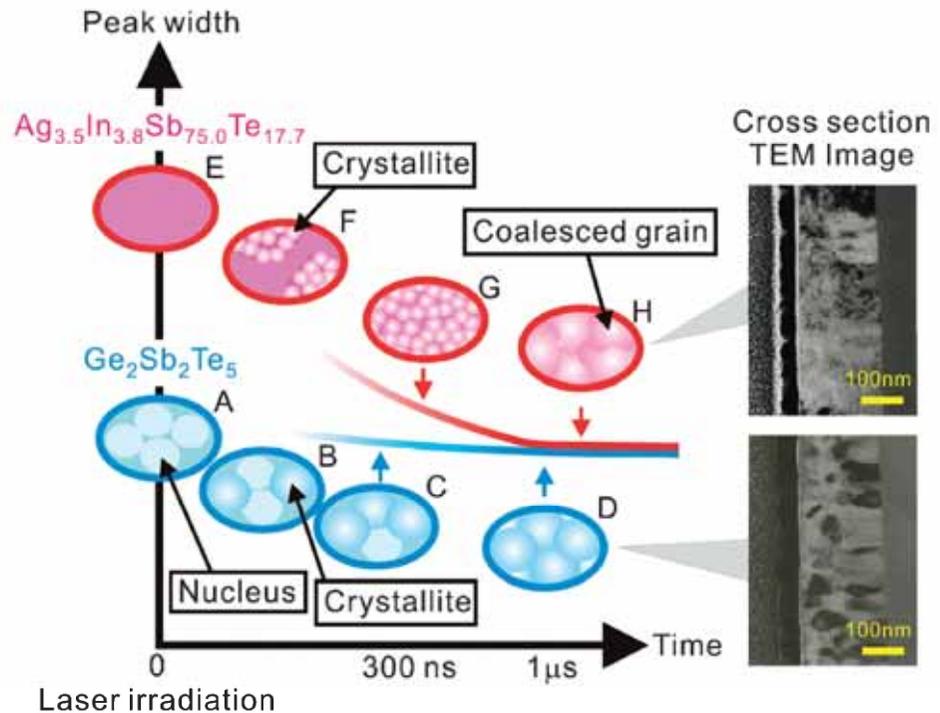
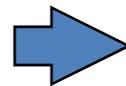
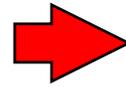
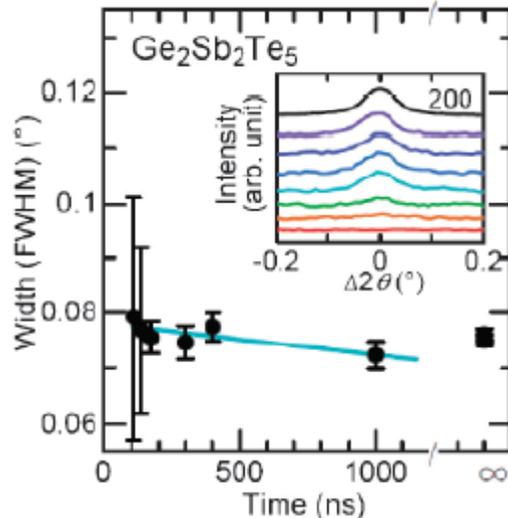
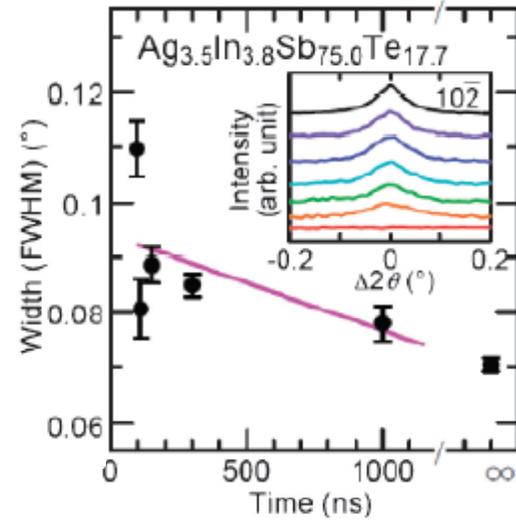
Crystal



# Movie of X-ray diffraction pattern for GST225



# Time-dependence of diffraction peak width (grain size and/or strain)



結晶成長過程の違い

Applied Physics Express, 1, 045001 (2008).

# まとめ

- DVD光記録材料におけるアモルファス-結晶相変化について、時間分解X線回折法を適用した。
1. 反射率と構造変化の強い相関
  2. AISTとGSTで結晶化過程に相違
    - ▶ AISTは結晶化過程でブラッグピーク幅が大きく変化
    - GSTはピーク幅は不変、(過渡的な巨大格子定数(予備データより))

# CREST研究(相変化材料の構造変化)関係者

高田昌樹(理研)

田中義人(理研)

大島隆(理研)

木村滋(JASRI)

小原真司(JASRI)

安田伸広(JASRI)

金廷恩(JASRI)

大沢仁志(JASRI)

福山祥光(JASRI)

守友浩(筑波大)

鳥海幸四郎(兵庫県立大)

松永利之(パナソニック(株))

山田昇(パナソニック(株))