Damage accumulation in thin ruthenium films induced by repetitive exposure to femtosecond XUV pulses below the single-shot ablation threshold

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Abstract: The process of damage accumulation in thin ruthenium films exposed to multiple femtosecond XUV free electron laser (FEL) pulses below the critical angle of reflectance at the Free electron LASer facility in Hamburg (FLASH) was experimentally analyzed. The multi-shot damage threshold is found to be lower than single-shot damage threshold. Detailed analysis of the damage morphology and its dependence on irradiation conditions justifies the assumption that cavitation induced by the FEL pulse is the prime mechanism responsible for multi-shot damage in optical coatings.

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1. Introduction

The development of high peak brilliance, high repetition rate free electron laser (FEL) light sources operating in the XUV and X-ray spectral regime such as FLASH, FLASH 2, European XFEL and LCLS leads to increased practical interest in the durability of thin films

exposed to a large number of pulses. FEL optical elements such as mirrors or beam-stoppers should be designed considering the limitation on durability to the FEL radiation together with requirements on high optical performance of the materials. Avoiding of FEL-induced damage is also critical when long exposures of thin film samples are foreseen during FEL experiments. Examples of such experiments can be found in time resolved studies of surface chemical reactions with pump-probe X-ray diffraction techniques [1].

Only recently the first papers on resistivity of metal films to multi-shot X-ray radiation were published [2, 3]. Earlier, Hau-Riege et.al. [4] investigated B_4C coatings. In both cases it was observed that the multi-shot damage threshold is slightly lower than the single-shot one, but no detailed analysis of the nature of multi-shot damage was yet reported. Within the extreme ultraviolet (XUV) wavelength range multi-shot damage studies of bulk Si samples were reported by Sobierajski et al. [5]. The prime damage mechanism was identified as crossing the melting threshold due to heat accumulation in the vicinity of the Si surface.

In this work we investigate the multi-shot damage threshold (MSDT) for Ru coatings exposed to different number of 100 fs pulses at 13.5 nm wavelength generated with different repetition rates. We present the dependence of the MSDT on the repetition rate and the number of pulses. The influence of irradiation conditions, namely the number of pulses and grazing incidence angle, on the morphology of the damaged spots is investigated. Mechanisms responsible for the observed damage phenomena are suggested, although detailed atomistic simulations are required to confirm the proposed mechanisms.

Understanding the multi-shot damage of Ru films has a practical relevance. Since Ru has a high reflection coefficient for the extreme ultraviolet and soft X-ray wavelength ranges, and has a low oxidation rate, it is a very attractive material for reflective coatings. This work is a continuation of a series of studies of optics durability exposed to FEL radiation below the single-shot damage threshold published here [6] and a detailed analysis of the nature of single shot damage discussed here [7].

2. Experimental

For this study, polycrystalline Ru coatings of 50 nm thickness were deposited on to super-polished silicon substrates using magnetron sputtering in Ar atmosphere. The thickness of the Ru layer was determined by X-ray reflectivity measurements. Exposure of the Ru films was performed at the BL2 beamline of the FLASH facility [8]. Details of the experiment can be found elsewhere [6, 9]. For irradiations we used 100 fs XUV pulses with a wavelength of 13.5 nm. The light was p-polarized with respect to the sample surface. We used the multi-shot irradiation mode where FLASH generated one pulse train per 0.1 second [10]. The maximum duration of a pulse train was 400 μs and the maximum repetition rate within the pulse train was 1 MHz. During the experiment we controlled the number of pulses within the pulse train by reducing the duration of the pulse train. It was also possible to reduce the repetition rate while keeping the pulse train duration constant, and thus reducing the number of pulses in the pulse train.

The determination of the MSDT was performed using Liu's method [11-13] and fluence scan method [9, 14] based on measuring damaged areas with differential image contrast (also referred to as Nomarski) microscopy. The dependence of the damage threshold on the repetition rate and the number of pulses was studied using irradiations at 3.17° grazing incidence with an effective area of the beam [14, 15] on the sample surface of 40546 μm^2. Development of the damage morphology was studied by analysis of damage craters caused by different number of pulses arriving at 20° grazing incidence at a fixed average fluence per pulse using a focused beam with an effective area on the sample surface of 120 μm2. The XUV reflectivity values at 3.17° and 20° degrees were measured to be 95.5% and 68%, respectively [16]. In both cases only the evanescence wave penetrates into the film resulting in a comparable penetration depth for these different angles of incidence - 2.5 nm and 3.5 nm,

respectively. For Ru, the critical reflection angle for XUV radiation at a wavelength of 13.5 nm is 27°.

For a consistent comparison of the damage threshold values at different irradiation conditions, all analyses is performed in terms of absorbed fluence. It is calculated according to the formula Fabs=(1-R)*E/A where E is the total energy of the pulse, A is the effective area of the beam on the sample surface determined according to [15], and R is the reflectivity coefficient at the corresponding exposure conditions. The total energy of the pulse E was measured with gas monitor detector described by Tiedtke et.al. [17].

3. Results and discussions

First, we present the results of multi-shot damage experiments of 50 nm Ru films performed at a grazing incidence of 3.17°.The Ru films were exposed to single pulse trains, consisting of a different number of pulses from 50 to 400 at a fixed repetition rate of 1 MHz. Another set of exposures was performed for a fixed number of pulses in a pulse train (100 pulses), but upon varying the repetition rate (time interval between pulses in a train). The measured damage threshold values are summarized in Table 1.

Table 1. Dependence of the multi-shot damage threshold in mJ/cm2 (absorbed fluence) on repetition rate and number of pulses in a pulse train at 3.17° grazing incidence. The error in the threshold determination is in the order of 20%.

Repetition rate, kHz	Number of pulses			
	50	100	200	400
250		7.5		
500		8.3		
1000	13.3	8.3	5.8	6.7

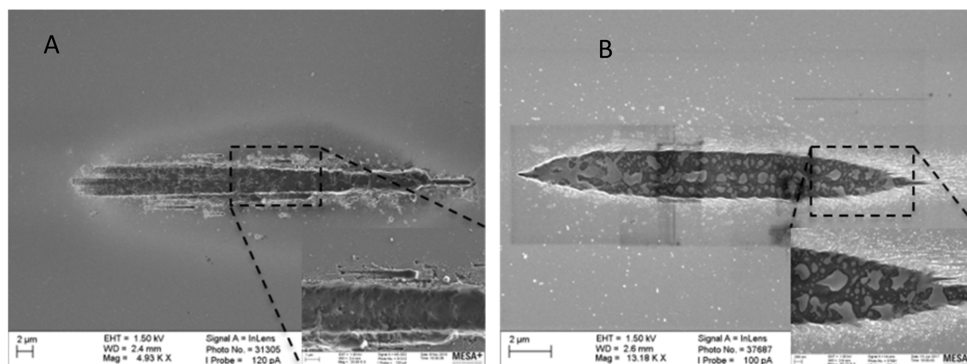

Fig. 1 HR-SEM image of a damaged spot produced with a) pulse train consisting of 50 femtosecond XUV pulses with 1MHz repetition rate at 3.17° grazing incidence angle and b) 400 femtosecond XUV pulse trains at 20° grazing incidence. Each pulse train consisted of 400 pulses. Pulse train repetition rate was 10 Hz, while pulse repetition rate within a pulse train was 1MHz.

As one can see, the MSDT does not depend on repetition rate within the error margin which in our case was around 20%. This suggests that heat accumulation is unlikely to be the damage mechanism for the given experimental conditions. This is in contrast to the

previously reported work of Sobierajski et al. [5], where heat accumulation in bulk Si exposed to similar radiation condition but at a normal incidence was found to be as the main mechanism responsible for multi-shot damage caused by XUV femtosecond FLASH laser pulses. Ru, being a metal, has a high thermal conductivity, which guarantees that the energy that is absorbed at the surface, diffuses through the entire Ru layer into the Si substrate mostly before the next pulse in the train arrives.

Another observation is the decrease of the damage threshold upon increasing the number of pulses to a fixed repetition rate of 1 MHz stabilizing at 200 pulses and more. This result suggest together with the independence of the MSDT on the repetition rate described above, that there is an accumulation of an irreversible process. Increase of MSDT with the decrease of the number of pulses from 100 to 50 indicates that damage caused by a smaller number of pulses with low fluence is not be observed, and that a large fluence of individual pulse is needed to cause detectable damage.

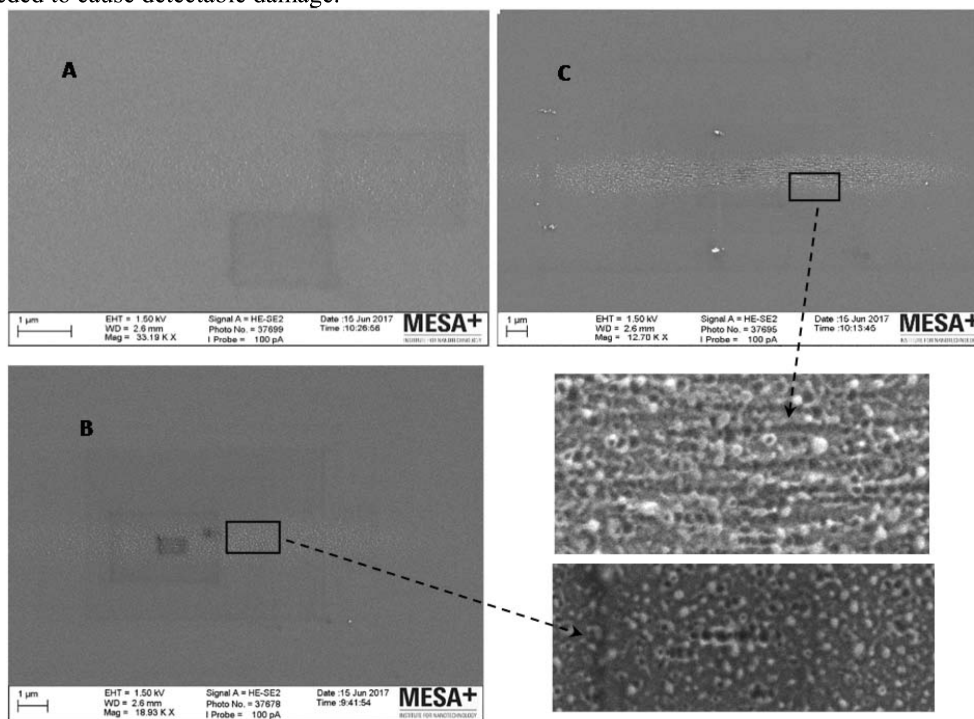

Fig. 2 HR-SEM images of damage morphologies caused by 1600 (A) 209 (B) and 9200(C) femtosecond XUV pulses at 20° grazing incidence condition.

In order to understand the processes responsible for the multi-shot damage of Ru, we performed ex-situ analysis of the damage morphology by means of high-resolution scanning electron microscopy (HR-SEM) and transmission electron microscopy (TEM). Additionally, we compared the damage morphologies obtained at 3.17° and 20° grazing incidence to study the influence of different angles of incidence. The HR-SEM image of a damaged spot exposed to a single pulse train that consisted of 50 pulses with 1 MHz repetition rate at 3.17° grazing incidence is shown in Fig. 1(a). The mean value of the fluence per pulse was 15 mJ/cm^2, which is close to the measured damage threshold value in table 1, which is 13.3 mJ/cm^2. The damaged spot has a total area of ~320 μm^2 and can be characterized as a deep crater penetrating through the Ru layer into the Si substrate. The fact that the crater depth exceeds the thickness of the Ru layer is verified by imaging, using an energy selective backscattered detector (not shown), which is sensitive to elemental composition of a surface,

confirming that Si is at the bottom of the crater. No other significant surface modifications around the craters, such as increased roughness or cracks, were detected by means of HR-SEM. Fig.1(b) shows a crater resulting from irradiation of the Ru film with a much larger number of pulses, namely 160 thousand (400 pulse trains of 400 pulses each at a repetition frequency of 1 MHz), but performed at 20° grazing incidence and a mean value of fluence per pulse of 12.5 mJ/cm^2. From the color contras we can conclude that the crater shown in Fig. 1(b) has smooth silicon surface with Ru droplets on top. Fig. 1 shows that, although both exposures are carried out with a fluence close to the damage threshold, a much smaller number of pulses arriving at 3.17° causes comparable damage to the one caused by pulses arriving at 20° grazing incidence.

To elaborate on the mechanism of accumulation of damage we study the development of damage with increasing number of pulses for irradiation at 20° grazing incidence angle. The following exposures were analyzed: 209 individual pulses generated at 5 Hz repetition rate, and 4 and 23 pulse trains, generated with 10 Hz repetition rate. Each train in this experiment consisted of 400 pulses generated with 1 MHz repetition rate. The damage threshold value, determined by Liu's method, for one such pulse train at 20° was 14±3 mJ/cm^2.

The mean value of the fluence per pulse in the irradiation was 12.5 mJ/cm^2 which is slightly below the MSDT. Only surface modifications of the Ru layer were detected (see Fig. 2). TEM and AFM measurements of the damaged spots (not shown) confirmed that only the Ru layer is modified. All three damaged spots presented in Fig. 2 can be characterized as increased surface roughness, although the degree of roughening is different. Surprisingly, the surface roughness is considerably lower after irradiation with 1600 pulses, than after 209 individual pulses, whereas the opposite is expected.

The possible explanation of this contradiction is the random character of the pulse fluences within the irradiation. We should note that because of the self –amplified spontaneous emission (SASE) operation of the FEL, the fluences of individual pulses varied from 1 to 30 mJ/cm2. The histograms of absorbed fluences per pulse for irradiations with 209, 9200 and 1600 pulses are shown in Fig. 3.

The solid lines in Fig.3 show the number of pulses within the exposure sequence that have a fluence higher than the x coordinate value. Based on such analysis (see the magnified image in Fig. 3) one can see that there are more high-fluence pulses (more than 20 mJ/cm^2 of absorbed fluence) in the case of irradiation with 209 pulses than in the case of 1600 pulses. If we assume that there is a damage threshold in the range of 20-25 mJ/cm^2 it will lead to the conclusion that with 209 pulses, the Ru film was exposed to a larger number of pulses that can cause damage than in the 1600 pulses case, explaining the larger damage with 209 pulses.

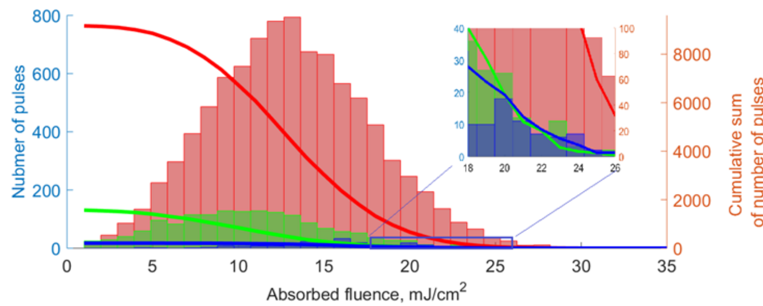

Fig. 3 Histograms of fluences for irradiation containing 209 (blue), 1600 (green) and 9200 (red) pulses at 20 degrees grazing incidence. The solid lines correspond to the cumulative sum of the number of pulses with a fluence equal to and higher than the x coordinate value calculated by the formula: $\sum_{F=F(x)}^{FMax} N(F_{abs})$, where F is the absorbed fluence, and N(F_{abs}) is the number of pulses with a fluence F_{abs}.

In our recent investigations [7] we showed that the nature of single-shot XUV-induced ablation of Ru is photomechanical spallation in the stress confinement regime. The latter means that heating of the lattice is faster than the acoustic relaxation time, which means that heating occurs at almost isochoric conditions. It was shown in damage studies of metals induced with optical lasers [18-20] that this situation leads to the generation of large thermo-induced stresses and, as a result, to spallation of the top part of a metal. The single-shot spallation threshold at 20° grazing incidence was measured to be F_{spall}=64+/-13 mJ/cm^2 of absorbed fluence, while the melting threshold was calculated to be F_{melt}=13 mJ/cm^2 [7]. It is known from literature [18], that spallation starts with nucleation of subsurface voids or cavities in a melted layer of irradiated metal, created as a result of propagation of a tensile stress wave. In a particular fluence range high enough to cause melting, but not sufficient to induce complete spallation, the cavities can remain frozen below the surface, which was proven experimentally [19-21] and with molecular dynamic simulations [19]. Therefore, for metals there is a cavitation threshold that is lower than the spallation threshold.

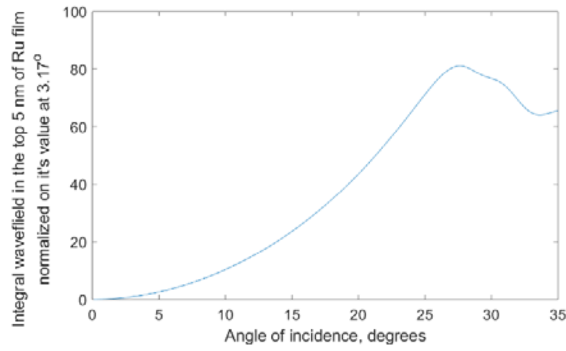

Fig. 4 The integral of the absorbed energy density in the top 5 nm of the Ru film on the angle of incidence, normalized to its value at 3.17°.

Within this damage mechanism, we suggest that individual pulses with the highest fluence in a pulse train are capable of not only melting the surface of Ru, but causing cavitation below the surface, as described above. The existence of subsurface cavities can create significant roughness and swelling of the surface [19-21].

This hypothesis can also explain the difference in damage caused by multiple pulses at 3.17° and 20° grazing incidence (Fig. 2). The angular dependence of the absorbed energy, simulated as the integral of the electromagnetic field (EM) in the top 5 nm of the film [22], normalized to its value at 3.17 ° is shown in Fig. 4. In the region of total external reflection, for Ru for 13.5 nm light ranging from 0 ° to 27 ° grazing incidence, only evanescent waves penetrate into the film [23]. The penetration depth in our case is about 3 nm . However, the density of the EM field increases with increasing angle of incidence and, therefore, the absorption dose also increases. The increased absorbed fluence may eventually reach the spallation threshold, resulting in locally occurring spallation of Ru.

The difference in the damage caused at very grazing and close to critical angles of incidence can be explained assuming the cavitation to be the onset of damage. The atomic force microscopy (AFM) image of the surface damaged by 209 pulses from Fig. 2(b) is shown in Fig. 5.

In a first approximation, the roughness observed in Fig. 5, can be considered as surface areas inclined at a certain angle θ* to the prime film surface. Therefore, the effective incident angle at the surface is not θ but θ+θ* and therefore the absorption dose is locally increased. According to Fig. 4, the same increment θ* of the incidence angle will cause much more increase in the absorbed dose for less grazing angles of incidence. For 3.17° prime incidence angle θ, an increase of the angle of incidence, for example by θ*= 20° as shown in Fig. 5, will

lead to an increase of the absorbed dose by a factor of 70, what for fluence of 15 mJ/cm^2 means locally an increase to 1 J/cm^2, that is much more than spallation threshold. However, for an incidence angle of θ=20° similar inclination will increase the absorbed dose by not more than 50%, leading to an effective maximum fluence of 45 mJ/cm^2 for the pulse with the highest fluence in the histogram in Fig. 3. This is still below the spallation threshold for a pristine Ru film.

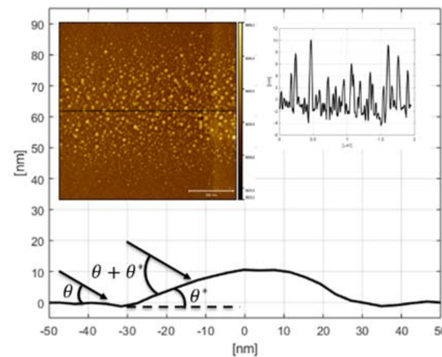

Fig. 5. The AFM image of damage caused by 209 pulses. The black line on the AFM image (top left) indicates the cross-section of entire scan shown in top right corner. The magnified cross-section of the height of one swollen region (bottom plot) shows that due to swelling the incidence angle can be increased by θ*=20°.

4. **Conclusions**

We present an experimental study of the damage caused by multiple ultra-short XUV pulses in Ru coatings. We found that the multi-shot damage threshold does not depend on the repetition rate by comparing damage threshold values obtained for 100 pulses arriving at 3.17° with repetition rate ranging from 250 KHz to 1 MHz.

Based on analysis of the development of damage morphology caused by an increase in the number of pulses arriving at 20° grazing angle and with a fluence close to the damage threshold, we suggest that the prime cause of the multi-shot damage is roughening of the Ru surface induced by a cavitation process. Therefore, multi-shot damage threshold should be equal to the cavitation threshold.

Comparing the morphology of the craters created pulses arriving at 3.17° and 20° grazing incidence we arrive at the conclusion that surface roughening should lead to local increase in the absorption of radiation, which may subsequently lead to local spallation of the roughened areas in Ru films.

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