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MICROMILLING OF TUNGSTEN CARBIDE USING FOCUSED ION BEAM

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ABSTRACT

This paper describes the effect of focused ion beam (FIB) micromilling parameters on tungsten carbide for the fabrication of any microcomponents. The FIB parameters such as aperture size, ion dose, dwell time, etc. were investigated in this study. A series of experiments were conducted on tungsten carbide with varying operating parameters to establish the correlation how the parameters affect the micromilling process and quality of the final component. All the experiments were carried out with dry micromilling using serpentine scanning mode. Empirical models were formulated to predict the sputtered depth which increased with higher ion dose significantly but the relationship was nonlinear. Some of the experimental results are discussed with qualitative judgement as it was difficult to explain quantitatively.

Keywords: FIB, Micromilling, Tungsten carbide.

1. INTRODUCTION

FIB micromilling offered the possibility of removing small quantities of target material without the use of photo resist or any other metal masking layers. In the micromilling process, the incident beam transferred sufficient momentum to the target surface and then the near-surface atoms escaped through cascades of collisions [1]. Initially this technique was mainly applied on silicon based materials such as integrated circuit repair and modification, maskless silicon micromilling, material analysis by SIMS (secondary ion mass spectrometry) technique, TEM (tunnelling electron microscope) sample preparation, etc. [2-4]. But recently, FIB micromilling had wide applications in non-silicon based materials such as nickel, tungsten carbide, steel to fabricate various microcomponents such as microcavity for microreplication, etc. [5, 6]. In addition, FIB micromilling was widely used for the fabrication of microcavity for replication of polymer microcomponents based on LIGA (lithography, electroforming, molding) principles [7]. Because of this increasing popularity of FIB micromilling of non-silicon materials, the effects of its operating parameters were essential to determine and optimize. Micromachining of metallic materials such as tungsten carbide was difficult when the target was to achieve micrometric accuracy and nanometric surface finish [8, 9].

2. EXPERIMENTS

The effect of FIB parameters were analysed by experiments. This included the micromilling of tungsten carbide sample with variable parameters and their investigation. All the experiments were carried out with dry micromilling using serpentine scanning mode. In this mode of scanning, the redepositions were almost uniform. A 50 keV dual focused e^-/Ga ion beam (Micrion 9500EX) integrated with scanning electron microscope (SEM) and energy dispersive x-ray (EDX) system (Oxford Link ISIS300) was used for this study. Geometry and surface characteristics of each of the fabricated microfeatures were investigated by using SEM and scanning probe microscope (SPM, Digital Instrument, Nanoscope IIIa). Before the detailed description of the micromilling experiments, the mechanism of FIB scanning and the related parameters are discussed first.

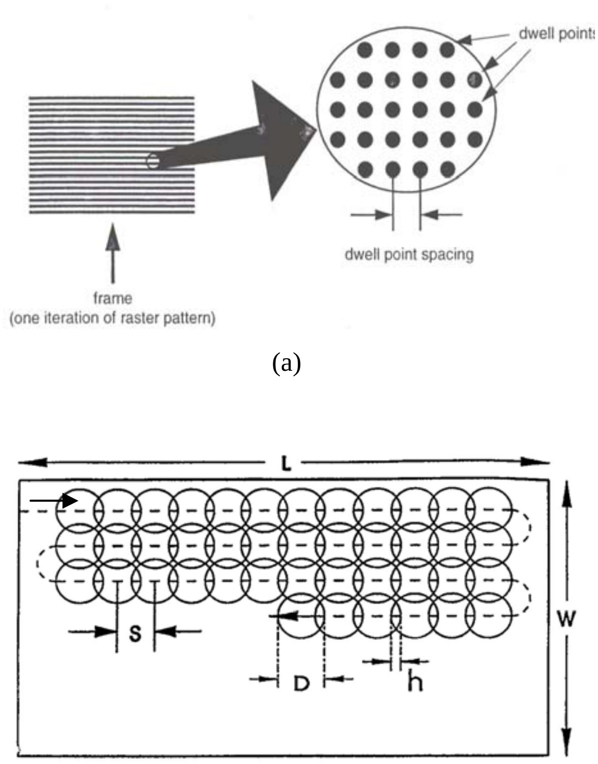
The FIB was guided by electrostatic lens to make the beam spot size as small as possible e.g., several nanometers. The FIB milled an area by deflecting from a point to another which was called rastering. The beam scanned in such a way that it traced a pattern of closely-spaced parallel lines as shown in Figure 1a. Each of the scan lines constituting the raster scanning pattern was in fact a linear array of discrete points to which the beam was rapidly deflected and at which it paused or dwelled for a predetermined period. These points were called dwell points. The length of time that the ion beam remained directed at the dwell point was called dwell time. There were two types of rastering. If the beam moved in the same direction for each of the scanning line then the scanning was called simple rastering. In this case beam must return to the initial state after finishing the previous line. The time required to come back to the original position was called retrace time. But after scanning one line, if the beam moved in opposite directions from one row to the next, the scanning mode was called serpentine scanning as shown in Figure 1b. This type of scanning eliminated the long retrace time from the last pixel of one row to the first pixel of the next row. In the following subsections, the effect of aperture size, ion dose, and dwell time are discussed in detailed.

2.1 Effect of Beam Limiting Aperture Size

It was assumed that the ion beam had a Gaussian profile, and its diameter was normally expressed as the full-width at the half the maximum (FWHM) height of the ion intensity distribution. With such distribution of ion intensity, more material was removed at the axis of the beam than around the periphery. The beam limiting aperture was like a punched hole to restrict the tail of the Gaussian profile. The aperture allowed only the central high intensity core of the beam to pass through and hence the aperture had a proportional effect on the spot size. The bigger the aperture, the bigger the beam spot size. For example, 75 μm aperture size could provide a 17 nm spot size. The FIB used in this study was equipped with six different apertures and these were 75μm, 100μm, 150μm, 250μm, 300μm, and 350μm. Experiments were conducted for all of these apertures. Square boxes of 10 μm x 10 μm size were micromilled using each of the above apertures for 5, 10, and 15 nCμm^{-2} ion dose. Other FIB parameters were 10 nm pixel spacing, and 10 μs dwell time. Each of the experiments was repeated twice. The milling time for each of the boxes was recorded during the milling processes. The etch depths and the surface textures were inspected and measured by SEM and SPM.

Experiments showed that changing in aperture size had negligible or no effect on the etched depth. The relationship of aperture size on the milling time is shown in Figure 2. It can be observed that longer milling time was required to deliver the same amount of ion dose using smaller sized aperture. However, the milling time did not increase proportionally but exponentially. Therefore, higher milling rates could be achieved by using a bigger aperture size. The aperture sizes not only affected milling time but also the surface finish. When milling using smaller aperture, i.e., very sharp beam, the surface texture become very clear and it resulted in poor surface finish. But the opposite things happened for the larger aperture size under the same other operating conditions which diminished the sharp texture by the tail energy.

A large beam resulted in the highest target current and the worst resolution. On the other hand, smaller aperture resulted in finer beam shape and lower target current, thus achieving smaller beam spot size. The energy distribution of the beam also broadened for the bigger beam spot size. Thus, the effect cause by the tails of the Gaussian beam became more severe and resulted in rounded edge, tapered walls, as well as unsatisfactory geometrical integrity. Therefore, it was concluded that higher milling rates could be obtained at the sacrifice of resolution. Considering this factors, a medium sized aperture, e.g., 150 μm, could be appropriate to achieve higher accuracy at reasonable milling time.



(a)

(b)

Fig 1. (a) FIB raster scanning in general, (b) serpentine mode of scanning (D = beam diameter, h = beam overlap, S = beam step size, L = length, and W = width of the milled area)

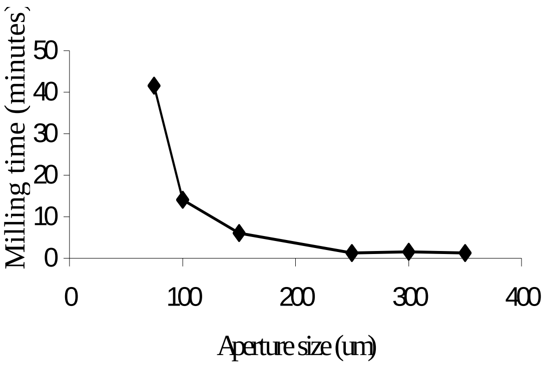


Fig 2. Effect of aperture size on milling time. (ion dose = 5 nCμm^{-2}, dwell time = 10 μs, pixel spacing = 10 nm).

2.2 Effect of Ion Dose

The ion dose was the quantity of ions delivered to the substrate per unit area. The higher the ion dose the higher the milling depth would be. The dose delivered for any particular milling was a function of the beam current, pixel spacing, dwell time, and the number of rasters. The units of dose was nanocoulombs per square micrometer (nCμm^{-2}). The sputtering yield in the form of number of substrate atoms per unit incident ion was readily available but it was for some specific materials and ion column. For the various FIB machine configurations, material properties, machining environment, etc., the sputtering yield was characterized and used as a reference for the machining rate. Experiments to determine the effect of ion dose was carried out by using 150 μm aperture, 10 nm pixel spacing and 10 μs dwell time. Square boxes of 10 μm x 10 μm size were micromilled with different ion doses. Then the milled depths were measured using SEM and SPM. The relationship between ion dose and milled depth was found to follow Eq. (1) which is a fit polynomial. This experimental equation was used for calculating the required ion dose to mill any desired depth on tungsten carbide substrate.

$$z = 0.2397\,d + 0.0115\,d^{2} \tag{1}$$

where z (μm) is the depth of micromilling using an ion dose of d (nCμm^{-2}) under dry micromilling condition (not gas assisted).

2.3 Effect of Dwell Time

FIB micromilling experiments for different dwell time were conducted. Square boxes of 10μm x 10 μm size were micromilled using 5 nCμm^{-2} ion dose and 10μm pixel spacing for the dwell time of 1, 5, 10, 50, 100, 500, and 1000 μs. Each of the milled boxes was examined under SEM *in-shitu*. For each of the cases, the number of raster, time per raster, and total raster time were calculated by the FIB software as listed in Table 1. The number of raster decreased as the dwell time increased. Shorter dwell time had shorter raster time per raster which was compensated by the increase in the total numbers of raster for each milling box. Finally, the total raster time remained the same for a particular ion dose and aperture size. Therefore, when the dwell time reached 500 μs and above, there was only one raster, which was the minimum.

One SEM micrograph of the seven milled boxes is shown in Figure 3. Although the total ion dose was the same for all of the milling boxes, long dwell time produced completely different results from short dwell time. Such results could be explained by the redeposition effect of the ion beam micromilling. Redeposition of material sputtered from the floor of the milled pit caused distortion of the wall profile. A slow single scan with high dose could result in tapered walls or even overhanging protrusions. It was obvious that this would not occur in the repetitive scan case.

Table 1: Relationship among dwell time, raster time and the number of raster.

Dwell time (μs)	Number of raster	Time per raster (sec)	Total raster time (sec)
1	460	0.46	210.07
5	92	2.26	207.65
10	46	4.51	207.31
50	10	22.54	225.41
100	5	45.07	225.37
500	1	225.34	225.34
1000	1	450.67	450.67

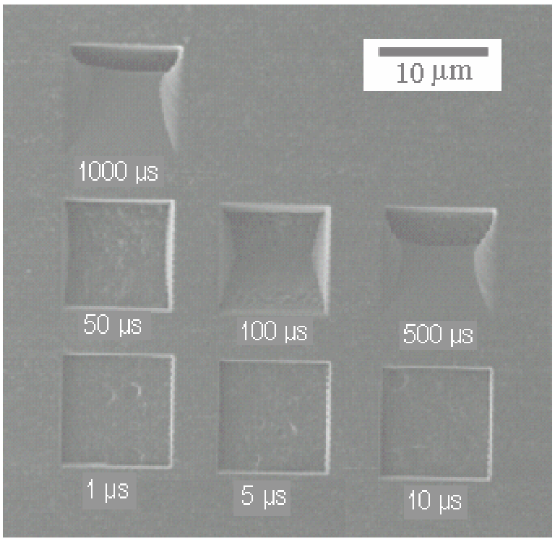


Fig 3. SEM micrograph of microstructures on tungsten carbide produced by FIB micromilling using different dwell time as indicated beside the milled boxes. (aperture = 150 μm, ion dose = 5 nCμm^{-2}, dwell time = 10 μs and pixel spacing = 10 nm).

Each such scan removed the redeposits built up from the previous scan to produce a clean wall profile. Due to this reason, the dwell time for FIB micromilling should be chosen within 1-10 μs for tungsten carbide substrate for a minimum redeposition. So, if one pass milling with long dwell time was substituted by repeated fast scans with shorter dwell time, redeposition could be substantially reduced.

3. CONCLUSIONS

Micromilling of tungsten carbide using FIB was studied by experiments. The effects of three influential FIB parameters were analysed. These were aperture size, ion dose, and dwell time. This experimental study showed:

1. FIB micromilling with bigger aperture sizes was faster than the smaller aperture sizes. But the bigger aperture provided low scanning resolution and hence the geometrical accuracy of the milled structure was poor. For bigger aperture, the energy distribution was broad and the spot size was bigger. It resulted in rounded edge, tapered walls, as well as unsatisfactory geometrical integrity. On the other hand, smaller

aperture resulted in sharper beam shape and smaller beam spot size. Therefore, higher milling rate could be obtained at a sacrifice of resolution. As a result, it would be better to select a medium sized aperture (e.g., 150 μm) as a compromise. The issue of surface finish was opposite. As the smaller aperture produces fine and sharp beam, the surface roughness was higher compared to bigger aperture size. But this issue did not affect the ultimate applications as the average surface finish was always within 2-5 nm.

2. Based on the experimental results, a fit polynomial equation, Eq. (1), was formulated for the calculation of micromilled depth for any ion dose. But this equation was developed considering a negligible redeposition (aspect ratio < 10). When the aspect ratio was more than 10, the redeosition dominated or exceeded the sputtering rate and hence Eq. (1) lost its validity.
3. Longer dwell time accumulated more redeposition and caused the final shape to be distorted. Shorter dwell time required more number of raster to deliver the required amount of ion dose. Because of such repeated scan, the redeposited materials partially eliminated. So, if one scan milling with longer dwell time substituted by repeated fast scan with shorter dwell time, redeposition could be substantially reduced. But regardless the shorter or longer dwell time, the total milling time remained the same if same aperture and ion dose were used. So, as the experiments showed, the dwell time of the FIB micromilling should be within 1-10 μs.

4. REFERENCES

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