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Suitability of electroless nickel for diffractive optics manufacturing

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Abstract
Diffractive optical elements (DOEs) are used in various applications, e.g. diffraction gratings for Raman and LIPS spectroscopy. The manufacture of DOE can be achieved by ultra-precision machining (UPM). UPM manufactured diffraction gratings are either shaped with rectangular diamond cutting tools or ruled, in which the grooves are formed by cylindrical diamond tools. Regarding higher production quantities, usually a master grating is manufactured and replicated. Therefore, the material of the master grating should provide high hardness and enable low roughness values. Electroless Nickel-Phosphorous (Ni-P) is widely used in UPM and offers those properties. However, the material-specific limits, machining strategies, and process parameters need to be determined for the two mentioned manufacturing processes.
For this purpose, experimental and numerical investigations were conducted and are presented in this paper. The examined groove widths are in the range of 1 – 10 µm. The experiments for ruling and shaping processes were conducted on a LT-ULTRA MMC 1100 ultra-precision machining center. The diffraction efficiencies of ruled gratings were calculated by rigorous coupled wave analysis. In order to determine the tool wear, cutting experiments of large-area gratings have been conducted. Manufacturing grooves with doubled width on predetermined positions enables to indirectly measure the cutting edge displacement of the tool in dependence of the cutting length by means of atomic force microscopy.
Opposed to other grating materials like Au, defect-free grooves could not be ruled into Ni-P. The grooves are not formed completely by low process forces whereas forming with higher forces lead to a removal of material in the upper part of the groove. This reduces the calculated diffraction efficiency by at least 26.3 % which is why further research in ruling of diffraction gratings in Ni-P is advised. However, much lower geometrical defects can be achieved by shaping the grooves. During the experiments, roughness values Rq ≤ 1.5 nm and cutting lengths l_c > 3 km without significant wear were achieved, which enables the manufacture of large, high-efficient DOE in Ni-P.

Microstructure, electroless Nickel, DOE, Ultra-precision

1. Introduction

There are several use-cases which demand high-efficient analytical equipment, e.g Raman spectroscopy for analysis of low-concentrated contaminants in liquid solutions. As the accuracy of these systems demands high-priced-quality components like diffraction gratings, prices are often not affordable for consumer-level appilcations.

One approach to reduce prices is to achieve lower manufacturing costs via replication of master-gratings. Manufacturing of curved as well as planar DOEs with high-efficient blaze-geometry is mostly done by means of lithographic/holographic processes [1]. These processes demand high effort in master-grating-production and are limited in form and structural dimensions of the blaze-geometries. An example for this are echelle gratings which contain groove widths and heights of several microns and are not manufacturable with current available laser wavelengths for holographic illumination.

One approach to reduce diffraction grating costs and expand the range of manufacturable DOEs is to alter the manufacturing process. By ultra-precision diamond machining each groove is generated sequentially, similar to the original diffraction grating manufacturing processes described by Rowland [2]. This enables a low radius of curvature as well as a large variety of grating groove geometries. In order to achieve large procuction quantities, the material selection must consider low levels of surface roughness and a sufficient hardness for a considerable amount of replication cycles via injection moulding or hot embossing. A replacement of the diamond tool within the manufacturing comes with finite precision in adjustment and therefore causes noticeable defects/form deviations of the DOE. This means that at least a finish cut must be conducted with one diamond tool. As damages of the diamond tool are geometrically projected into the groove geometry, the tool wear behaviour, in particular chipping and cutting edge displacements, should enable a production of surfaces with sufficient size.

Diamond cutting is widely used to machine electroless nickel (Ni-P), as the amorphous microstructure of the material enables low achievable roughness values. Ni-P alloys with amorphous microstructure can be deposited in an autocatalytic process [3]. However, as two different machining processes with only partially known suitability for shaped Ni-P gratings [4] are used, suitable process parameters and technical limits are partially unknown. In order to investigate the achievable geometrical and optical qualities, various experimental and numerical investigations have been conducted which are discussed in this paper.

2. Experimental setup

The experimental investigations have been conducted with an ultra-precision machining center LT-ULTRA MMC 1100. The machine tool is modified by additional stiffening, additional housing for thermal stabilisation, vibration decoupling and 2 additional rotary axes.

Two machining processes have been investigated: ruling and shaping depicted in Figure 1. For the ruling process cylindrical diamond tools are used which were mounted to a flexure-based tool holder which enables a precise adjustment of the ruling force F_r. For the shaping experiments rectangular shaped monocrystalline diamond tools have been used. A summary of the applied process parameters is given in Table 1

Table 1: Process parameters of experimental investigations

Process	Shaping	Shaping (tool wear)	Ruling
Grating period b	1 µm	5 µm	1 µm
Blaze angle α_b	6°	1 °	5 °
Diamond tool	HPHT	MCCD, HPHT	MCD, rwz = 6mm
Material/Workpiece	100 µm Ni-P coating		
Cutting/ruling speed v_c/v_r	1000 mm/min	1000 mm/min	550 mm/min

A Nanosurf Nanite atomic force microscope (AFM) was used for the measurements of surface topologies. Larger-scale measurements were conducted on a Zygo Niewview 5015 White Light Interferometer (WLI). The examined samples were brass substrates with amorphous, high phosphorous content Ni-P coating. A more detailed description of the used experimental tools and devices is given in [5]. The diffraction efficiencies were numerically calculated by rigorous coupled wave analysis (RCWA).

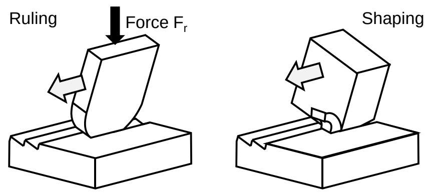

Figure 1. Ruling and shaping (schematic)

3. Experimental results

3.1. Ruling

The ultra-precision ruling of diffraction gratings is an adaption of the original mechanical manufacturing process and is mostly carried out on thin Au layers. In order to determine suitable process parameters for amorphous Ni-P coatings, experimental investigations have been conducted. Moreover, the resulting theoretical diffraction efficiencies have been calculated for the experimentally generated grooves and compared to those of the nominal, desired geometry. In the ruling experiments, two ranges of parameters, groove width $1.0\ \mu m < b < 1.8\ \mu m$ and blaze angle $3° < \alpha_b < 5°$, were investigated.

By ruling the structures with low ruling force F_r, an underfilling occurs, wich means that the groove facets, especially the upper sections, are not completely formed by the ruling tool and therefore do not correspond to the tool geometry. However, with the raise of the ruling force F_r, a removal of material in the upper part of the grating grooves is caused.

This ruling behaviour differs to Au [5]. It is assumed, that the removal at high ruling force F_r is caused by a brittle behaviour of the Ni-P in which parts of the material breakup during the forming process. This assumption is supported by high amounts of particles observed on the grating surface. This means that no microstructures free of geometrical defects are achieved in the parameter range by ruling in Ni-P coatings.

In Figure 2 an AFM-profile measurement of a ruled diffraction grating and the corresponding calculated diffraction efficiencies for various diffraction orders under orthogonal illumination are displayed. It can clearly be seen that there is a significant geometrical deviation between the nominal/desired and the measured groove profiles. A calculation of the corresponding diffraction efficiencies is displayed in the lower section of Figure 2. The difference in diffraction efficiency in first diffraction order $\Delta\eta_{m1}$ amounts an absolute value of 14.08 %. As the diffraction efficiency of the nominal grating profile is $\eta = 31.32$ %, this means that there is a relative loss of first order diffraction efficiency of 55.02 % which is caused by the geometrical deviations of the measured grooves. Similar results have been observed for other geometries, with a minimum loss of 26.3 %. A variation of ruling speed did not show any siginificance to the results which means that the ruling force F_r is the only variable parameter. As there is no ruling force F_r for production of low-defect Ni-P-DOE in the examined parameter range ruling is considered to be not suitable for production of high-efficient DOEs in the investigated parameter range on amorphous 100 µm thickness Ni-P coatings.

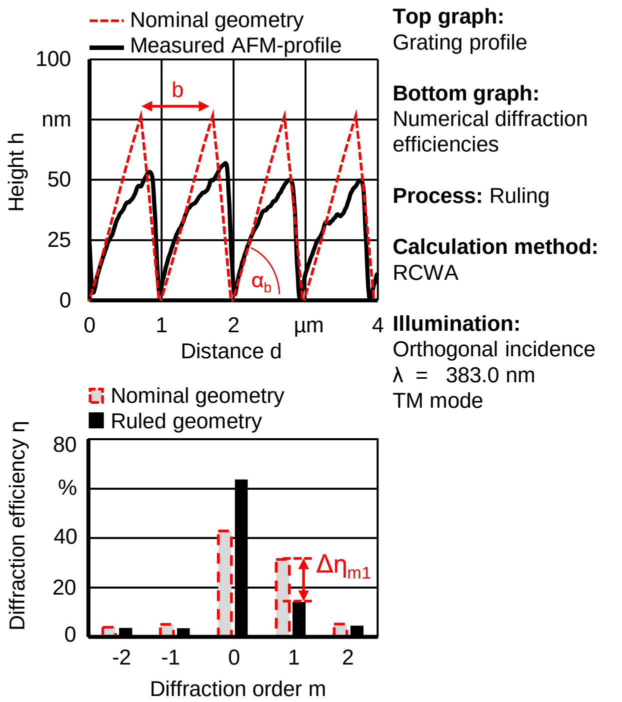

Figure 2. Ruled Ni-P-Grating and corresponding calculated diffraction efficiencies

3.2. Shaping

While amorphous Ni-P coatings are commonly used in ultra-precision machining, the potential geometries and limits in the manufacture of blazed DOE yet need to be determined.

For this purpose shaping experiments regarding achievable geometry, roughness values, and cutting lengths have been conducted. The cutting tools used were type Ib synthetic monocrystalline diamonds manufactured by high pressure high temperature processes (HPHT-MCD). As seen in Figure 3, the 1st order diffraction efficiency in TM-Mode of the shaped groove geometry is much closer to that of the desired geometry with a relative loss of 13.03 %. It is assumed, that this remaining loss is mainly caused by the roundings of the groove edges. A comprehensive investigation of theoretical and measured diffraction efficiencies for shaped gratings remains to be conducted. Yet, the depicted grating poses a challenge for cutting processes and still enables efficiencies with only half the loss of best achieved ruled gratings. This indicates that much lower relative losses can be achieved with larger shaped grating geometries where the edge roundings contain a smaller share of the geometrical profile.

Opposed to ruling the observed geometrical deviations were significantly lower in the shaping experiments. Few to no defects were observed for groove widths 1 µm ≤ b ≤ 10 µm, depicted in Figure 3.

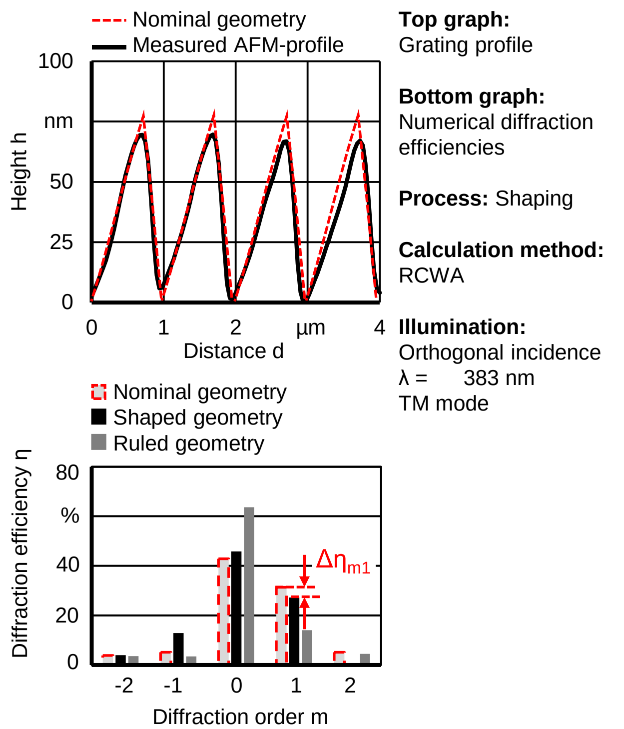

Figure 3. AFM-profile measurement of diffraction grating with 1 µm grating period

The diffuse scatter of an optical surface depends on the RMS-surface roughness. Therefore, the RMS-roughness Rq has been determined by WLI-measurements of planar test gratings manufactured with varying cutting depths a, cutting speeds v_c, and rake angles γ. The roughness Rq is determined from extracted line profiles along the groove direction. The results can be seen in Figure 4.

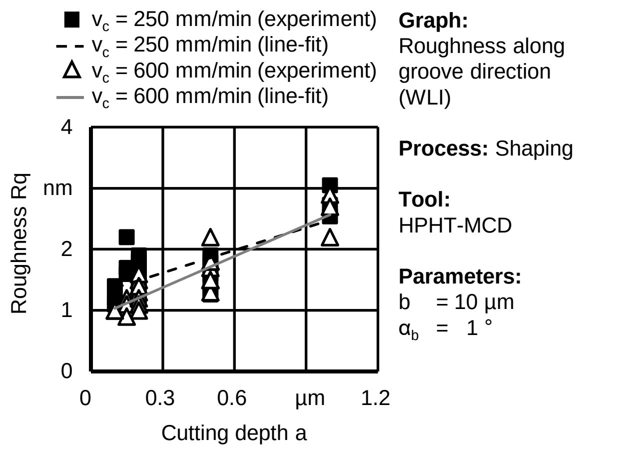

Figure 4. Roughness Rq in shaped Ni-P-Structures for various cutting depths a

As the roughness Rq correlates to the cutting depth; minimum values of Rq < 1 nm have been achieved for cutting depths a ≤ 200 nm. However, as the samples are pre-machined by fly-cutting and/or milling, a minimum nominal cutting depth of a ≥ 0.5 µm is advised to ensure low cutting depth variations. Depending on the groove geometry multiple cuts might be necessary in order to achieve a finish cut with a low cutting depth 1 µm > a > 0.5 µm.

4. Tool Wear

For production of larger surface areas, tool wear is crucial. As each groove is cut consecutively, abrasive tool damages are transferred into the groove profile [4]. As the groove positions are defined by the trajectories of the cutting edge, any cutting edge displacements lead to a groove dislocation. Especially cutting edge displacements towards the rake face cause a dislocation of the grooves and therefore a form error of the DOE. In order to determine the tool wear of rectangular shaped diamond tools in DOE manufacturing, cutting experiments with cutting lengths 2 000 m < L_c < 7 000 m have been conducted.

A precise quantitative measurement of cutting edge displacements in the single-digit nanometer range on a diamond cutting edge poses a difficult task for even SEM or AFM measurement devices. In addition a direct diamond tool measurement after certain cutting lengths L_c would demand a disassemble and therefore cause a readjustment of the tool which always comes with finite accuracy and would therefore alter the cutting depth after each diamond tool measurement. Therefore, the cutting edge displacement was measured indirectly on the groove profiles of experimental shaped DOE. For this purpose grooves with doubled groove width b have been cut on predefined positions, respectively cutting lengths L_c. Figure 5 displays an AFM-profile measurement of a double-width groove for different cutting lengths L_c.

It can be seen that the blaze facet contains a unsteady section which resembles the form of the grooves with nominal groove width b = 5 µm. This corresponds to the abrasive wear towards the rake face of the diamond tool which can be determined by measurement of the width of the damaged section.

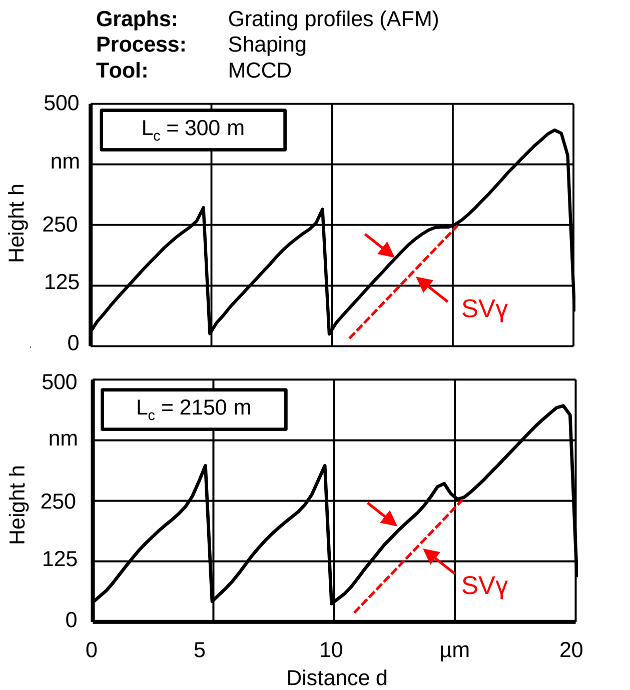

Figure 5. AFM-profile measurement of diffraction grating with 1 µm grating

The measured cutting edge displacements SVγ are depicted in Figure 6. The most noticeable effect is the massive abrasive wear on the CVD-deposited diamond tool (MCCD) which is roughly one order of magnitude larger than that of the type Ib HPHT diamond tools. For all HPHT diamond tools a maximum cutting edge displacement SVγ < 4 nm has been observed which is reached after approx. 2 000 m < L_c < 4 000 m. This can be considered as a limit of manufacturable cumulated groove length of diamond shaped Ni-P-DOE. However, as the graphs indicate an asymptotical progress much larger cumulated cutting lengths can be achieved, if cutting edge displacement SVγ < 4 nm can be tolerated in the DOE design.

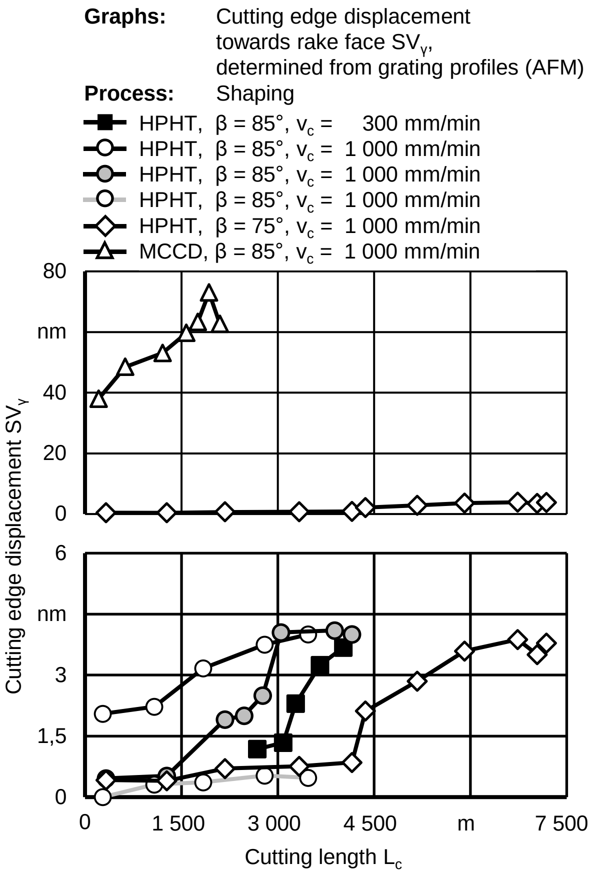

Figure 6. Cutting edge displacement towards rake face SV_γ in dependance of cutting length L_c for various diamond tools

Another possible type of tool damage is the occurrence of edge chipping [6]. This would cause irregularities on the blaze facets and therefore cause additional stray light and/or reduce diffraction efficiency. However, during the experiments no cutting edge chipping has been observed on any of the examined blaze grooves, respectively diamond tools.

5. Large surface DOE

In order to demonstrate the machinability of large-surface DOE a series of three echelle-gratings with a surface of each 50 x 50 mm² have been manufactured. With a groove width f = 13.33 µm, each grating contains a cumulated groove length of 765 m. As expected, no significant tool wear has been observed.

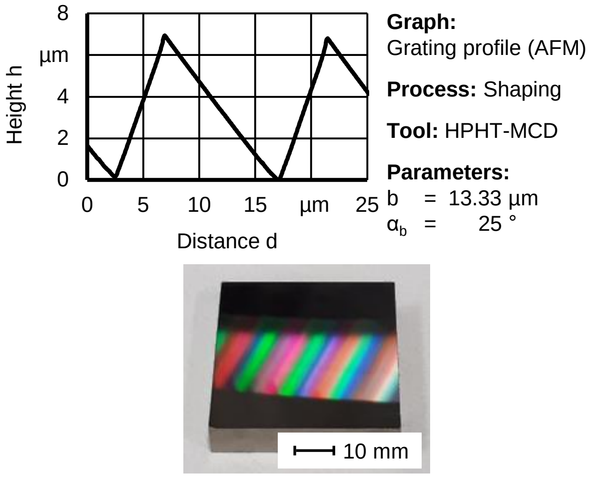

Figure 7. Echelle grating in Ni-P

6. Conclusions

Within this paper various experimental and numerical investigations regarding diamond-machined Ni-P DOE are presented. Ruled gratings show more or less severe groove defects which could not be eliminated. Optimal results still correspond to diffraction efficiency losses of at least 26.3 %. Therefore it excludes ruling for manufacturing Ni-P DOE, unless geometries and/or process parameters are found which enable less geometrical defects. Shaped DOE with so far minimal achieved groove widths b ≥ 1 µm contain much less geometrical defects.

A roughness of Rq < 1nm is feasible at extremely low cutting depths but larger cutting depths a > 0.5 µm should be applied in the real/final process. Consequently, a roughness of Rq > 1.5 nm should be expected in the DOE shaping. Synthetic type Ib HPHT diamond tools enable cutting lengths L_c > 2,000 m wich was demonstrated by manufacture of three 50 x 50 mm² echelette gratings.

Future activities will focus on further investigations of the influence of diamond-type on tool life and wear behaviour, longer cutting lengths as well as profund theoretical and experimental investigations of shaped grating diffraction efficiencies.

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