euspen's 16th International Conference & Exhibition, Nottingham, UK, May 2016
www.euspen.eu

Surface reproduction accuracy of holographic design features for machine components fabricated by a combination of TiN-physical vapour deposition and a specific subsequent galvanic process

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Abstract
Based on a combined plasma based and galvanic process, an ultra-hard surface structure embossing tool will be developed to stamp holographic design features for example in embossing roller for the print and packaging industry as well as dies for striking coins or machine components. The multilayer bond, respectively the mutual multilayer adhesion is investigated, discussed and supported by parameter studies and evaluated by adhesive tape tests. Comparative investigations pertaining to surface reproduction accuracy of holographic design features are carried out using arc evaporative physical vapour deposition (PVD) and magnetron sputtering. The physically deposited titanium nitride(TiN)-layers are compared by their particular layer composition quality, deposition rate N and layer adhesion. In summary, holographic structures can be replicated in hard coatings with high surface reproduction accuracy. In comparison to the Arc-PVD process, the highest accuracy can be reached by magnetron sputtering.

Keywords: titanium nitride, hard coating, PVD-coating, hologram

1. Introduction

True color hologram printing is widely known from consumer goods industries concerning packaging, labeling or holographic embossed coins. Apart from design and colour aspects, there are functional aspects of holographic design features as well. It can be used to maintain the uniqueness of a certain product. A major challenge in this regard is the embossing of holographic design features in steel components as usually used in the machinery and plant engineering sector. For this purpose, an ultra-hard surface structure embossing tool will be developed to stamp holographic design features in machine components.

While the fabrication of holographic structures in the range of visible light is state of the art, the replication of this holographic structure with high surface reproduction accuracy in hard coatings, such as TiN is a new approach. The structure height of the holographic foil is approximately $400\ nm \le h_l \le 600\ nm$. The original basic holographic master matrix material is still covering the tool surface. In order to achieve the final embossing tool surface, it needs to be separated or delaminated from the TiN-coated holographic embossing structure without damaging the TiN-layer. The use of such an ultra-hard TiN-layer extends the overall embossing tool life and therefore reduces manufacturing costs.

2. TiN-coating

Titanium (Ti) is highly inert at room temperature ϑ_{RT}. It reacts with nitrogen to TiN at temperatures $\vartheta > \vartheta_{RT}$ or in plasma. TiN is characterized by its high hardness H, wear and chemical resistance.

A change of colour from silver to gold for stoichiometric TiN is caused by the integration of nitrogen in the Ti-grid. In case of a high nitrogen content, the colour appears to be red. The resistance against deformation, delamination and breakage is significant for stamping shallow holographic structures into hard substrates. Breakage of the ultra-hard micro structured surface layer is avoided because of a specially modified and supporting underneath galvanic multilayer tool construction. The intended hard layer system consists of at least one TiN-hard layer and three dampening Ni-layers of declining hardness levels. TiN, as a ceramic hard material, has a high Mohs hardness $H_M = 9$ and is chemically stable at the same time [2]. Stoichiometric TiN has a material density $\rho \approx 5.3\ g/cm^3$. The material density ρ of TiN rises with increasing nitrogen content [3]. An additional online deposited Ti-layer simplifies the following nickel(Ni)-plating to set up the embossing tool since TiN is classified as inert with reference to galvanic processes.

3. PVD-coating technology

TiN can be fabricated with different PVD processes. It is described how the different TiN-coating PVD-technologies, magnetron sputtering and Arc-PVD, influence the surface reproduction accuracy of holographic design features. A distinction of how the material is transferred into the gas phase for deposition can be drawn between

- vaporization by means of electron beam, laser or resistance-heated source of evaporation and
- plasma-enhanced technologies (e.g. sputtering)

3.1. Arc evaporative physical vapour deposition

Arc-PVD uses an electric arc to deposit vaporised material from a cathode target on a substrate. The Arc-PVD process is particularly suitable for the deposition of nitrides and carbides, such as TiN, Ti and a wide variety of metals and alloys [4]. The deposition rate N and the coating temperature ϑ_C is comparatively high in comparison to sputtering [5].

3.2. Magnetron sputtering

DC-magnetron-sputtering, as special variation of the conventional sputtering, allows a higher deposition rate N because of the additional magnetic field. The secondary electrons, emitted from the cathode, are circling in fixed cycloid orbits near the target surface. The increased retention time t_r leads to a higher degree of ionisation and hence to an increased deposition rate N. However, magnetron sputtering benefits from a significantly lower process pressure ρ with reduced substrate heating. The term "reactive sputtering" of TiN is used when nitrogen gas is introduced into the plasma chamber in addition to the argon (Ar) plasma [4].

4. Experimental study

The substrate, a thin holographic aluminium (Al) foil, has a thickness $d_s \approx 100$ µm and dimensions A = 5 mm x 5 mm. Different machining and cleaning processes were used for the preparation of the aluminium substrates. Arc-PVD and magnetron sputtering have been studied and compared to assess the surface reproduction accuracy of TiN-coated aluminium holograms (see fig. 1).

Figure 1. PVD-coating machines – a) for magnetron sputtering: Leybold Heraeus Z400, b) for Arc-PVD: Interatom PVD 20"

Table 1 shows the parameters used for coating processes a) Arc-PVD and b) magnetron sputtering.

Table 1 Process parameters for a) Arc-PVD, b) magnetron sputtering

a) Arc-PVD	
Parameter	**Value**
Current I [A]	70
Bias voltage U [V]	150
Reaction gas N_2 [sccm]	100
Deposition rate N [nm/min.]	≈100

b) Magnetron sputtering	
Parameter	**Value**
Mode	DC
Power P [Watt]	150
N_2 pressure p [mbar] Ar pressure p [mbar]	2E-4 4E-3
Deposition rate N [nm/min.]	≈35

Substrate surface-related contamination, e. g. the dense natural aluminium oxide layer with a thickness of 1 nm ≤ d_s ≤ 100 nm [5], must be taken into account.

The surface condition of the substrate will be determined by cleaning in acetone to remove contaminants from the surface. Afterwards, a 5 minutes ultrasonic cleaning is carried out followed by rinsing the substrate with isopropanol and drying with nitrogen. The physically deposited TiN-layers are characterised regarding their particular layer composition, adhesion and surface design.

The possibilities on TiN-coating on a holographic aluminium foil (see fig. 2) are limited to the heat-sensitive substrate material and the substrate hardness H. The aim is to keep the coating temperatures ϑ_c as low as possible in order to not affect the holographic structure adversely.

5. Conclusion

As shown in figure 2b and d, the Arc-PVD TiN-coated foil exhibits temperature-related deformations. Light microscope images (LMI) with magnification factors up to 100x confirm the surface reproduction accuracy of magnetron sputtered TiN-coated holographic aluminium foils and state the considerably higher quality than of Arc-PVD fabricated TiN-layers.

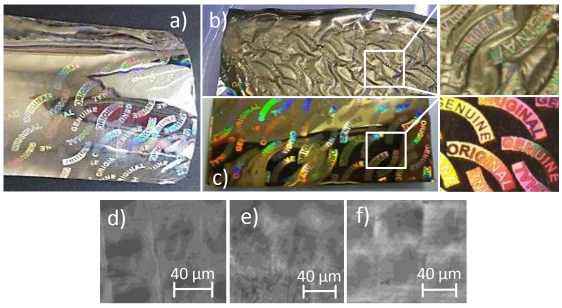

Figure 2. Quantitative comparison of both PVD-coating processes; a) 5 mm x 5 mm aluminium foil with hologram structure; b) Arc-PVD coated foil; c) magnetron sputtered foil; LMI of d) aluminium foil with hologram structure, e) Arc-PVD coated foil and f) magnetron sputtered foil

According to figure 2c the holographic lettering is still clearly readable after magnetron sputtering a TiN-layer with a thickness $d_s \geq 1$ µm (see fig. 2c). Precise layer thickness specs in the submicron level are difficult to set within the Arc-PVD process in contrast to magnetron sputtering processes. The surface quality of a magnetron fabricated layer is identically equal to the holographic substrate.

The average temperature ϑ_{DC} during magnetron sputtering is 70 °C ≤ ϑ_{DC} ≤ 100 °C and during Arc-PVD considerably higher, reaching 300 °C ≤ ϑ_{Arc} ≤ 650 °C, according to in-situ measurements. The significantly higher operating temperature ϑ_C leads to a loss of hardness H according to micohardness measurements on a fischerscope®. The Arc-PVD TiN-coating Vickers hardness is H ≈ 1,400 HV due to the high temperature load, similar to Lugscheider et. al. [5], whereas the magnetron based TiN-coatings show Vickers microhardness values of H ≈ 1,900 HV.

Essentially, the dense natural aluminium oxide layer does not have noticeable effects on the coating adhesion in both cases as demonstrated by the adhesive tape test results of the go/no-go type. The adhesion of Arc-PVD TiN-layer is not universally guaranteed in contrast to the magnetron sputtered TiN-layer according to the most recent status.

The authors wish to acknowledge the financial support provided for this research project by the Federal Ministry of Economics and Technology (BMWi).

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