Journal of Materials Processing Tech. 249 (2017) 221–228

Contents lists available at ScienceDirect

Journal of Materials Processing Tech.

journal homepage: www.elsevier.com/locate/jmatprotec

Drawability and size effects for micro-arrayed deep drawing of Ni-Co/GO nanocomposite foils

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ARTICLE INFO

Keywords:
Micro-arrayed forming
Micro deep drawability
Nanomaterials
Metal-matrix composites (MMCs)
Size effects

ABSTRACT

For the first time, three different male dies (a rigid male die, an aluminum male die, and a nanoceramic powder male die) are used for a micro-arrayed deep drawing process using graphene oxide (GO) reinforced Ni-Co nanocomposite. Micro-parts with different shapes can be formed using the different male dies, and models to predict crack positions are established. The nanocomposite size effect leads to an increased depth-diameter ratio for decreasing female die diameters, and the conservation of volume leads to higher depth-diameter ratios and thinning rates. The reinforcing GO phase not only is able to improve the micro formability of the composite, but also inhibits grain growth at high temperature.

1. Introduction

Recently, demand has been growing for micro-parts, particularly for micro-arrayed parts. Using the micro-arrayed forming method, complex shaped products can be produced with large quantities and low costs for many fields, such as biomedical, chemical analysis and microsystem technologies. Plastic micro-manufacturing can produce micro-metal parts by using plastic forming methods, and it has many advantages, such as easy processing, high productivity and low costs (Saotome and Iwazaki, 2001). This method is especially suitable for the mass production of micro-arrayed parts.

Micro-deep drawing is a micro-plastic forming method that can be used in metal foil forming and can produce many micro-parts of different materials. Ghassemali et al. (2013) successfully manufactured micro-pins with diameters of 0.3, 0.5, and 0.8 mm without any defects. Gau et al. (2013) fabricated micro-cups with large CH/OD (cup height/cup outer diameter) ratios using the austenitic stainless steel alloy 304. Vollertsen et al. (2010) achieved the micro-deep drawing of an alloy aluminum-scandium foil with a limit drawing ratio of 1.6. Due to the changing forces between the workpiece and die due to the miniaturization of parts, size effects occur with the new grain sizes, which can lead to difficulty in controlling the accuracy and high damage rate of the formed parts. Vollertsen (2012) used size effects to explain the changes in the forming behavior of foils with respect to the forming limit, which was denoted by the limiting drawing ratio. Abazari et al. (2015) explained and predicted the importance of micro- and nano-systems by using theoretical models. To decrease the influence of size effects and obtain better plastic formability, more grains should be contained in the micro-parts, and this is required to achieve a small grain size. Nanocrystalline materials have relatively small grain sizes and high mechanical properties, which can be applied in micro-plastic forming. Sène et al. (2011) investigated micro-deep drawing experiments for single crystals and polycrystalline aluminum foils and determined, displacement curves for the punch, directional changes of the grains and entire deformation curves. They also verified that the accurate critical strain value depends on the grain size, annealing temperature, and crystal orientation.

Composite materials can be prepared by adding a reinforcing phase to the matrix. Simões et al. (2015) and Karamis et al. (2004) found that the grain size of the matrix can be decreased and the mechanical properties can be improved by using this method. Liang et al. (2015) researched the morphology of grain size, and indicated that the reinforce effect of a flake-like phase with a large aspect ratio and specific surface area is better than a granular and fibrous reinforcing phase. Graphene (Gr), first found by Geim and Novoselov (2007), is a flake-like material consisting of a single layer carbon atoms with an SP^2 valence and a six-sided honeycomb shape. Changgu et al. (2008) measured the tensile strength and Young's modulus of Gr to be 130 GPa and 1 TPa, respectively. The mechanical properties of graphene oxide (GO) are similar to graphene and uses Gr and GO as reinforcing phases, which can improve the plastic formability. Dong et al. (2014) used laser sintering to achieve a single layer of GO reinforced composites using a Fe matrix. From this, surface hardness improved by 93.5%, and the fatigue life improved by 167%, indicating that GO could be used as a reinforcing phase in a composite to improve mechanical properties.

In this paper, Ni-Co/GO nanocomposites foils were used as original

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http://dx.doi.org/10.1016/j.jmatprotec.2017.06.002
Received 3 May 2017; Received in revised form 29 May 2017; Accepted 2 June 2017

materials for micro-arrayed deep drawing experiments, and the formability of the micro-parts was investigated. Different formabilities for a rigid punch, aluminum punch and nano-ceramic powder punch were investigated. The shapes of the formed parts, stress/strain, thickness distribution and cracked position were studied, and the height-diameter ratios for the different dies, thinning rates and grain size changes were also examined to determine the influences of size effect on the plastic forming process for nano-composites.

2. Experimental details

2.1. Materials

The material used in this study is Ni-Co/GO composite foil prepared by pulsed electrodeposition, similar to Zhang et al. (2008) and Chung et al. (2008). The plating bath contained 0.8 mol/L of Ni $(NH_2SO_3)_2{\cdot}4H_2O$, 0.007 mol/L of $NiCl_2{\cdot}6H_2O$, 0.5 mol/L of H_3BO_3, 0.1 g/L of sodium dodecyl sulfate, 1 g/L of saccharin and 0.05 g/L of GO. The thickness of the prepared foils was approximately 50 μm, and they were cut into a circular shape with a diameter of 15 mm for micro-deep drawing experiments.

2.2. Micro deep drawing tests

A 5 × 5 micro-arrayed die was used in this experiment. The diameters of the single hole were 400 μm, 600 μm and 800 μm. The distances between two adjacent holes' centerlines were 1.2 mm, 1.2 mm and 1.5 mm. The diameter of a single hole was 600 μm for the different female dies used in the deep drawing experiments. The rigid punch was a 5 × 5 micro-array with a diameter of 340 μm and a length of 2 mm for a single pin. The distance between the centerlines of two pins was 1.2 mm, and the single gap was 80 μm for the female die and rigid punch. The female die and punch material was high temperature resistant stainless steel manufactured by precision machining technology. Fig. 1a shows an overall die image for the rigid male die. The material used for the soft punch was 5083 aluminum with a thickness of 2 mm. For the micro-arrayed soft punch experiment, the foil covered the surface of the female die, and the aluminum sheet was placed on the foil. Next, the punch was placed on the sheet of aluminum. The aluminum soft punch flowed into the die with the foil each time, so the aluminum soft punch had to change for every experiment. An overall image of the aluminum soft punch is shown in Fig. 1b, and a schematic diagram of the micro-arrayed soft punch deep drawing device is shown in Fig. 1c. For the nanoceramic powder punch, Zr_2O_3 powder (with a granularity of 50 nm) was placed on the surface of the 2-mm-thick foil. Then, the rigid punch was placed on the nanoceramic powder. A schematic diagram of the micro-arrayed nanoceramic powder experiment equipment is shown in Fig. 1d.

The micro-arrayed deep drawing experiments were conducted on an Instron-3343 universal testing machine equipped with a resistance heating furnace. The test temperature was 500 ± 1 °C, and the drawing speed was set to 0.02 mm/min.

2.3. Characterization

The morphological characteristics were obtained using transmission electron microscopy (TEM, Tecnai G2F30) and scanning electron microscopy (SEM, Helios nanolab 600i). The thickness distribution was obtained using a metallurgical microscope (OLYMPUS GX51-F) and the three dimensional shape and size were measured using laser scanning confocal microscope (OLYMPUS OLS3000).

3. Results and discussion

3.1. Basic properties of the Ni-Co/GO foil

Fig. 2 shows a TEM image and electron diffraction pattern for the electrodeposited Ni-Co/GO composites. Fig. 2a and b shows TEM images of the Ni-Co/GO composites. According to Fig. 2a, the grains of the prepared material are uniform and have a small size of approximately 30–50 nm. Fig. 2b clearly indicates that GO exists within the composite (as shown in the box) and covers and connects to the Ni-Co matrix. Fig. 2c shows the electron diffraction pattern and XRD image. The electron diffraction pattern indicates that the prepared material is polycrystalline and that the diffraction peaks from the XRD are coincident with diffraction rings. The main crystal surfaces for the (111), (200), (220), (311) and (400) planes are also observed.

Fig. 3a shows the spectra from an energy dispersive spectrometer (EDS) for the fracture surface of the Ni-Co/GO composite. As shown, Ni, Co and GO are uniformly dispersed on the fracture surface at a mass percentage ratio of 97:2.3:0.6, which is similar to the amount in the plating bath. The proportions of these elements and their uniform distribution at the surface are necessary to ensure the good mechanical properties of this composite. Fig. 3b shows the surface SEM image; the surface is flat and silky with a relatively high density. The good surface morphology is beneficial for a micro-arrayed deep drawing process to ensure that the crack rate can be reduced and achieve better formability.

3.2. Micro-arrayed deep drawing experiments with different male dies

Fig. 4 shows the parts formed using different male dies, including a rigid male die (a), aluminum male die (b) and nanoceramic powder male die (c). From these images, all of the micro-arrayed parts are successfully formed when using these three male dies. The bottom is relatively flat when using the rigid male die. The formed parts are hemispherical when using the aluminum male die. The shape is irregular when using the nanoceramic powder male die. The differences in a single micro-part are minor between the rigid male and aluminum male dies, whereas there are significant differences in the height and shape achieved when using the nanoceramic powder male die.

The shape of the formed part is important to evaluating the deep drawability. Fig. 5 shows SEM images of single samples when the rigid male die (a), the aluminum male die (b) and the nanoceramic powder male die (c), respectively. According to these images, when using the rigid male die, the bottom of the formed part is flat, and the slopes of the two sides are similar to the frustum of a cone. When using the aluminum soft punch, the bottom of formed part takes on a regular semi-circular shape. When using the nanoceramic powder male die, the slopes of the two sides are different, and the symmetry is poor.

The rigid male die is cylindrical. During the deep drawing process, the bottom contacts the foil directly, and the bottom becomes relatively flat. There is a difference between the male and female die sizes. The rate of deep drawing is uniform, which means that the shape of the formed part is regular with similar slopes on the two sides. The melting point of aluminum is 660 °C, and the deep drawing temperature is 500 °C. At this temperature, the aluminum has relatively better formability. With the addition of the external force, the aluminum can provide force similar to that of the male die, and the foil will flow to this space. Simultaneously, due to the surface tension of the aluminum and internal attractive forces, the aluminum will be nearly spherical, so the formed part is relatively smooth and regular. The powder male die is composed of nanoparticles, which are small and easily to be aggregated. When using the nanoceramic powder male die, the number of powder particles in the die is random, resulting in the irregular shape.

The thickness distribution of the deep drawn parts has a significant influence on the forming quality. Fig. 6 shows a single part's thickness distribution and a metallograph of the cross-section for the rigid male