

# Influence of Stamp Forming Parameters on Final Part Properties of Hydroxyapatite Filled Ethylene Vinyl Acetate Co-Polymer Composites

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**SUMMARY:** Hydroxyapatite (HAP) filled ethylene vinyl acetate co-polymer (EVA) composites are potential candidates for craniofacial applications. A cost effective technique for obtaining these composites in the clinically significant forms would indeed be a landmark accomplishment. Stamp forming is one of such processes where the cost as well as the performance of the product strikes the right balance. This study was carried out prior to the stamp forming process optimization of the composites into 3-D contours, essential for applications like cranioplasty. In particular, experiments with regard to the development of a two-dimensional stamping method for processing of HAP filled EVA composites using an angle mold (90°) were carried out. The processing conditions like the stamping temperature, time, and stamping rate required to give high-quality right angle bends were established. The quality of stamped forms was gauged in terms of physical appearance, shape conformance and variation in wall thickness. It was found that the stamping temperature and velocity were the key factors, which determined the quality of the stamped part. Too high temperatures, and stamping rates led to severe thinning and degradation of the formed parts, while too low temperatures and stamping rates did not conform the composite to the mold contour. A processing window in terms of stamping velocity and stamping temperature was also established.

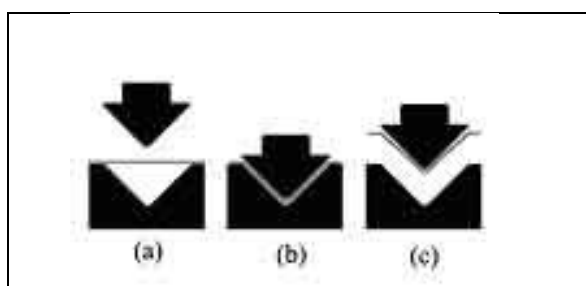
**KEYWORDS:** composite; hydroxyapatite; part quality; polymer; stamp forming, two-dimensional.

## INTRODUCTION

Hydroxyapatite (HAP) filled ethylene vinyl acetate co-polymer (EVA) composites are developed in an attempt to formulate “surgeon friendly” material for renovating impaired skull contours. The bone-bonding ability of HAP and pliability of EVA makes the composite ideal for this purpose. In cranioplasty, pre-fabrication of cranioplastic implants using computerized 3-D imaging and automated milling are employed to reduce the operating time and to obtain excellent cosmesis. These techniques, however, are expensive and require a long manufacturing time. Stamp forming has proved to be very cost effective method that improves the performance/cost balance of reinforced polymeric composites via high-speed manufacturing and shaping process [1]. In the current study, we report about the influence of stamp forming parameters on final part properties of HAP-EVA composite forms fabricated using an angle mold (90°).

## MATERIALS AND METHODS

Composite sheets having 40 vol.% HAP were used for the study. HAP for the preparation of the composite was synthesized by precipitation route involving ammoniated calcium nitrate and dihydrogen ammonium phosphate. The precipitated HAP was then converted into powder form by spray drying in a laboratory size mini spray drier (Buchi Mini Spray drier, B-181, Switzerland). Cryogenically ground EVA powder (Shriswasan Chemical (M) Pvt. Ltd., Mumbai, India) with particle size less than 300  $\mu\text{m}$ . The polymer matrix contained 28 wt.% vinyl acetate content and a melt flow index (MFI) of 25. The melting temperature of the matrix was determined by differential scanning calorimetry as 68°C. Rectangular composite strips of dimensions (130 x 30) mm<sup>2</sup> were cut from the 2.3 mm thick composite plates, heated above the softening temperatures in a hot air oven and stamp formed using a right angle tool fitted in the 80 kN stamp forming press (HY-Power OP 2MI-TR8-115/30, Italy). The forming technique is schematically represented in Fig. 1 and the variables used for forming are given in table 1.



**Fig. 1.** (a) Pre-heated composite sheet placed over cold female die, (b) sheet formed by male punch into female die, and (c) punch withdrawn and material free to spring backward.

**Table 1.** Processing variables for forming

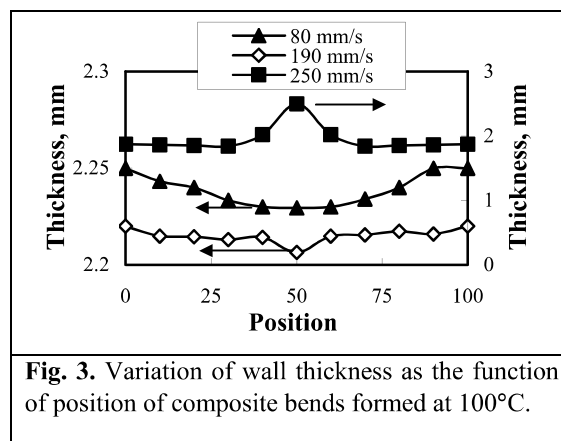
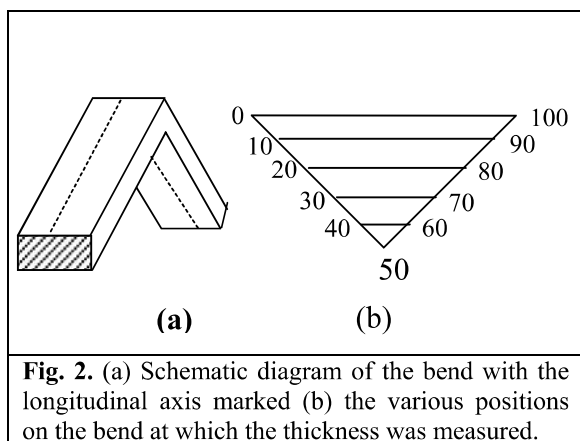
Tool temperature, °C	25
Pre-heat temperature, °C	80-120
Pressure, MPa	6
Forming velocity, (mm/s)	80-250
Time at pressure, (sec)	60

The quality of stamped forms was gauged in terms of physical appearance, shape conformance and variation in wall thickness.

## RESULTS AND DISCUSSION

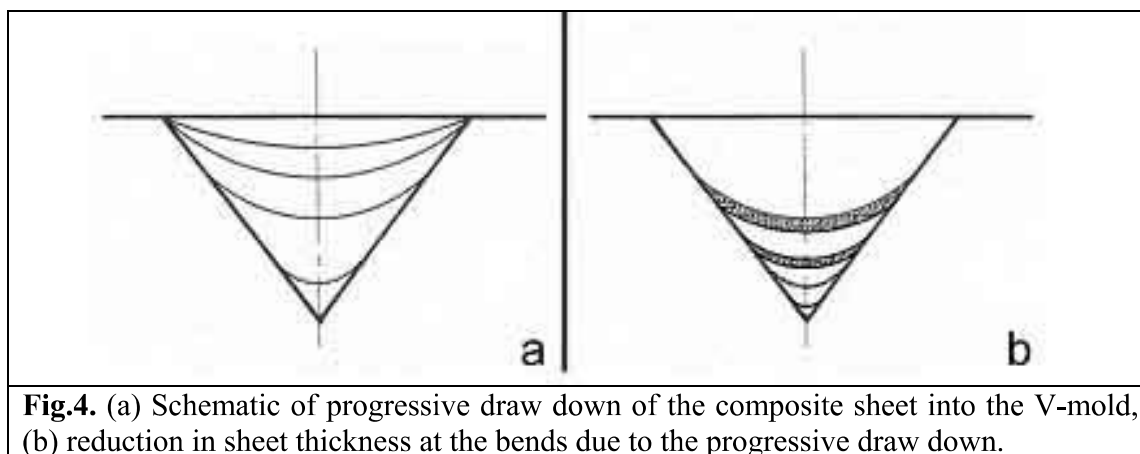
Uniform thickness distribution, optimum average thickness and shape conformance are important properties for manufacturing high quality stamped parts economically. One of the key parameters to obtain a part of a good quality is the selection of the pre-heating conditions [2]. The material has to be heated to a point where it is pliable enough to be shaped while supporting its own weight during the heating/transporting process [3]. Since the hot laminate is exposed to a lower environmental temperature before mold contact and deformation take place, the use of a sufficiently high closing speed also plays an important role in successful forming. In this investigation, two pre-heating temperatures, i.e. 80, and 100 °C were employed. Heating the composite sheets above 100 °C led to excessive sagging and difficulty in handling of the sheet. A series of trial runs were performed in which the stamp velocity was varied in the range 80-250 mm/s. The stamped composite bends thus obtained were examined for the uniformity in part thickness and part angle formed.

For measuring the variation in the thickness of the formed bend, different positions were marked along the longitudinal axis of the bend (Fig.2 a & b) and the thickness at these positions were measured with the aid of a micrometer. As an example, the thickness variation as a function of position for stamp formed bend at 100°C pre-heating temperature and various stamping velocities is given in Fig. 3.



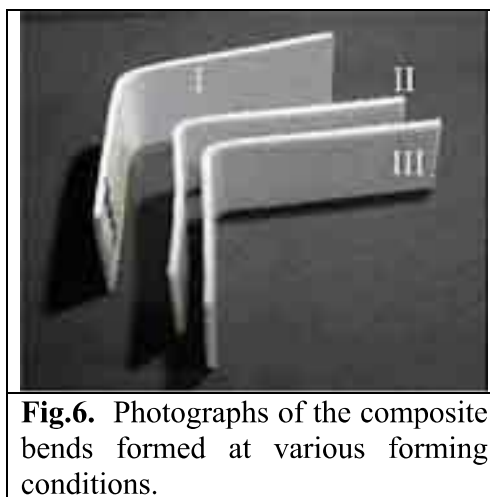
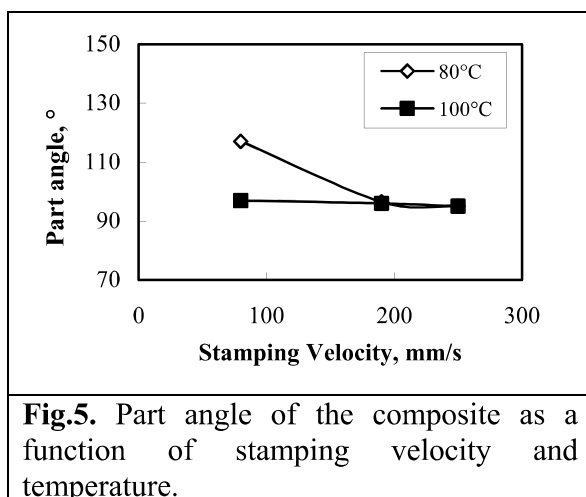
It is apparent from the figure that the quality of the formed bend is influenced by the variation of both the stamping temperature and stamping velocity. The thickness of the bends decreased with increase in stamping velocity. The nature of the thickness distribution curve for the bends formed at 80 mm/s and 190 mm/s stamping velocity was found to be similar. The formed parts had thinner walls at the bend and thicker at the sides of the V-bend. The average thickness of the part formed at 80 mm/s was, however, slightly higher than that formed at 190 mm/s. The thickness distribution of the composite formed at 250 mm/s had thicker walls at the bends and thinner towards the sides.

Stamp forming involves a complex mixture of material deformation processes and most forming processes do not yield parts having uniform wall thickness [4]. The variations in wall thickness of the formed contours have been found to be dependent on temperature and mode of deformation [5]. The observed variation in the wall thickness of the formed bend in the present study is the consequence of progressive draw down of the flat composite sheet into the mold (Fig. 4a).



During the initial stages of stretching, the sheet contacts the closest flat surface of the mold while other areas of the sheet continues to stretch. The part of the sheet that touches the cold mold in the initial stages of forming cools down while the other areas of sheet that has yet to touch the mold is still hot. This leads to a non-uniform temperature distribution over the sheet under deformation, resulting in dissimilar material deformation histories. At the bends the sheet contacts the mold during the remote stages of forming operation. The sheet, in this area, thus undergoes biggest stretching and/or largest deformation resulting in the smallest wall thickness at the bends (Fig.4b). At 250 mm/s, the temperature of the sheet at the time of stamping is quite high. The high sheet temperature leads to the material stretching more, enabling more material to be pulled further down the mold resulting in incidence of a web formation at the bends.

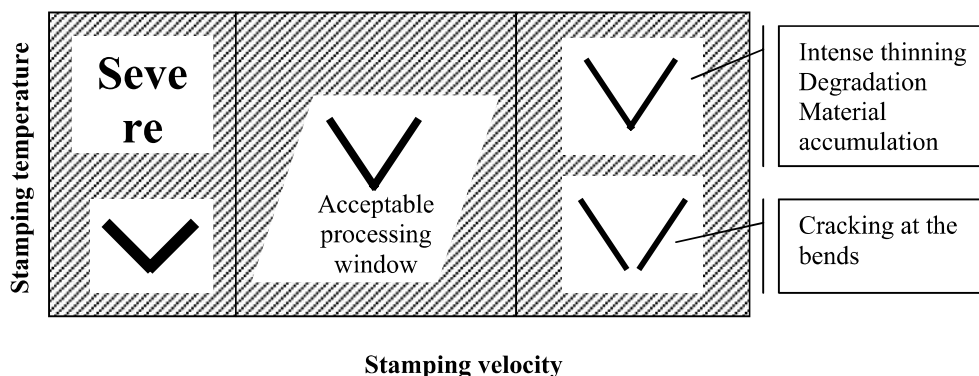
The variation of part angle of the composite with respect to the stamping velocity and temperature is given in the Fig. 5.



It is apparent from the figure that the measured angles of the formed bends are greater than 90°. This phenomenon is referred to as ‘spring back’ effect and results from the anisotropy of thermal properties of composite materials [6]. When the composite cools from the processing temperature to ambient temperature, the polymer matrix would contract more than the reinforcement HAP [ $\alpha_{EVA} = 160 - 200 \times 10^{-6}/^{\circ}C$ ,  $\alpha_{HAP} = 13 \times 10^{-6}/^{\circ}C$ ]. This leads to the development of residual thermal stresses within the composite bend. The stresses distort elastically, thus resulting in some degree of spring back. The formed angle is also influenced by the processing parameters employed. For example, severe distortion of the angle was observed when E28H40 was formed at 80mm/s stamping velocity and 80°C pre-heating temperature (I in Fig.6). The measured angle in this case was found to be 117°. Too low temperature and stamping velocity cools down the composite sheet making it stiff. As a result the sheet does not stretch easily and does not faithfully replicate the mold details. The formed angle decreased to 96° (II and III in Fig. 6) when formed at 100°C and 190 mm/s stamping velocity. This may be attributed to the more sufficient resin flow and the wider melted range at high working temperature.

## CONCLUSIONS

HAP filled EVA composites could be successfully formed into two-dimensional contours with the aid of stamp forming technique. From the trials performed a schematic diagram of the processing window for 2-D forming of HAP-EVA composites could be evolved (Fig.6).



**Fig. 7.** Schematic representation of processing window for 2-D stamp forming of HAP-EVA composites

The experimental studies showed that for successful forming, the temperature of the sheets has to be maintained in the range 90-100°C. This requires the composite to be heated to at least 20°C above the melting point of the polymer matrix. The forming velocity could vary between 190-250 mm/s depending on pre-heating temperature set. Too high temperatures (>100°C) led to severe sagging of the composite sheet, making it difficult to handle and transport. When stamped at high velocities this resulted in intense thinning of the walls and web formation at the bends due to accumulation of the material. Too low temperature and stamping velocity on the other hand cools down the composite leading to the formation of distorted forms.

## ACKNOWLEDGEMENTS

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