NANOINDENTATION HARDNESS AND MACROSCOPIC MECHANICAL BEHAVIORS IN FILLED ELASTOMERIC NANOCOMPOSITES

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Key words
Carbon black, Elastomer, Nanoindentation, Multi-scale behavior.

INTRODUCTION

Carbon black (CB) filled semi-crystalline ethylene butyl acrylate (EBA) copolymer networks are investigated to probe for the CB particles dependence of the deformation behavior from nano-to micrometers length scales of samples which are submitted to nanoindentation characterization. With respect to this purpose, the phenomenology for hardness ($H$) response in these materials indicates a typical increase of the hardness by decreasing the indentation depth ($h$) similar to the observed behavior in elastomeric materials. This behavior can be related to the change of the mesostructure, formed by the heterogeneous three-dimensional interconnected network of polymer and of aggregates of CB particles. Furthermore, The CB amount is found to increase the resistance of composite under the action of a mechanical stress. The $H$-$h$ curves were then compared to some analytical models and correlated to a tensile macroscopic behavior in order to highlight the involved deformation mechanisms with length scale. A complementary set of characterizations such as profilometry and atomic force microscopy probes were also employed to best understand of those mechanisms.

EXPERIMENTAL

In this study, we used an EBA copolymer filled by acetylene CB (Denka Black). The average size of the carbon black particles is 30 nm and the mean size of the primary aggregates is of order 150 nm \cite{1}. The specific surface depends strongly on the size of the primary particles is 63 m$^2$.g$^{-1}$. The nanoindentation hardness experiments were performed by using Berkovich method (Nano indenter XP MTS) with a force resolution of about 50 nN and displacement resolution of about 0.1 nm. For all experiments, the load were fixed in the range of 0.8-1.4 mN with a loading rate of 0.05 s$^{-1}$. For this study, different samples were prepared containing CB in various volume ratios from 7% up to 20%.

RESULTS AND DISCUSSION

Our motivation is to quantify the CB effect on the nanoindentation hardness and corretate the results with tensile experiments carried out at macro-level. Firstly, atomic force microscopy images provide us useful information about the well dispersion state of CB throughout the polymer matrix as is reported in the figure 1 by measuring the electrical resistance at nanometric scale. A typical hardness-indentation depth curve is depicted in Figure 2 for a sample containing 10.84 vol\% of CB.
indicating changing deformation mechanisms with the length [2]. \( H \) increases by factor of about 10 for the indentation depth range of 0.01 - 2.0 µm. In addition, the ultimate tensile strength (UTS) is found to increase linearly with average hardness for CB volume fractions (\( \phi \)) ranging from 7 to 20 vol % (not shown here) [3].

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{AFM_image.png}
\caption{A representative AFM image of sample containing 8 vol% of well dispersed Carbon Black.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{Hardness_Indentation.png}
\caption{A typical Hardness-Indentation depth sample containing 10.84% of Carbon Black.}
\end{figure}

CONCLUSION

This contribution deals with the nanoindentation characterizations of elastomeric filled nanocomposites. The deformation behavior at different length scales is examined according to the CB fraction volume and compared to some analytical models. A correlation is found between hardness and results tensile performed at room temperature. Other macroscopic properties such as glass transition temperature of the composite samples are linked to the nanoindentation data.

References