Asian Journal of Engineering and Technology (ISSN: 2321 – 2462)
Volume 02 – Issue 02, April 2014

Hardness Behaviour of Thermoplastic Cattle Horn using Nanoindentation Technique

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ABSTRACT--*The current paper study the hardness behaviour of Kano cattle horn under nanoindentation and micro hardness tests. Five different samples were generated and level A, B, C, D and E cut along the whole length of the horn from the skull to the tip respectively. The samples were indented by diamond Berkovich indenter in sub microns for the nano hardness test. The Vickers micro hardness test was performed using a Wilson Wolpert Vickers micro hardness machine. The results showed that the value of hardness is increasing with the increasing length of the horn. The hardness increased from the skull to the middle of the horn length and starts decreasing gradually to the tip of the horn. Both tests confirmed that the hardness value is approximately higher at the middle of the horn. The range of hardness values of sample A, B, C, D and E obtained using nanoindentation results are 0.24-0.32 GPa, 0.24-0.28 GPa, 0.21-0.24 GPa, 0.22-0.27 GPa and 0.07-0.14 GPa respectively. The hardness value obtained using Vickers micro hardness was 24 VH, 20 VH, 25 VH, 26 VH and 26 VH for A, B, C, D and E respectively.*

Keywords— Thermoplastic composite material, Cattle horn, Hardness property, Nanoindentation

1. INTRODUCTION

Natural horns are thermoplastic and can be heated, bent and shaped to create wonderful tools and useful things like horn handles, buttons, horn belt buckles, horn drinking vessels as well as horn fashion accessories based on requirements. Horns are naturally grown items and as such will have considerable variation between individual pieces. Requirement of specific sizes or shapes can be handled by the manufacturers (Baggot, 1988). The cattle horn is abundant in Nigeria, especially in the northern part, but the utilization is very low compared to what is obtained in other developed countries. The bulk of our horn was being exported to these developed countries, while a very little proportion is used in making things like chalk. The remaining is burnt in rubbish dumps and used as fertilizer. Many countries are now imposing import restrictions on "Animal Products" (which includes 'raw horns') to prevent the spread of infectious diseases. This increased restriction leaves us with the problem of how to dispose of the horns, in addition to the serious loss of revenue. Based on that, the researchers think of characterizing the horn and studying some of its mechanical properties for appropriate use in various field of engineering among which hardness property was recognized. Hardness is one of the most important properties among the mechanical properties of any engineering material. This important property can be determined using different ways, among which the most common one is the Vickers micro hardness testing. It measures the hardness of the material at micro and sub microns. Nano indentation is another way of testing the mechanical properties of the material at nano scale and it became a primary and attractive technique for determination of the mechanical properties of thin films and small structural features. The main feature of nanoindentation is that, it facilitates the measurement of properties such as hardness, modulus of elasticity etc, at both the micrometer and nanometer scale. Mechanical properties of nano scale materials can be determined directly from indentation load and displacement measurements without the image of hardness and used in the characterization of mechanical behavior of the materials. According to nanoindentation theory, hardness of the material can simply be defined as the mean pressure exerted by the indenter at maximum load and can be expressed as:

$$H = \frac{P_{max}}{A} \quad (1)$$

where P_{max} is the maximum load applied during the indentation and A is the projected area of contact between the indenter and the specimen. The measurements of indentation moduli and hardness depend on knowing the contact area of the indentations. For an ideally sharp Berkovich indenter, the cross-sectional area in terms of contact depth is expressed as follows:

$$A(h_c) = 24 \cdot 5h_c^2 \quad (2)$$

Where $h_c = \frac{A}{24.5}$ (3)

The use of animal horns by humans is controversial, especially if the animal was hunted specifically for the horn. This is because some animals are threatened or endangered to reduce populations partially from pressures of such hunting. Some people use bovid horns as musical instruments. Bovid horns removed from the bone core, cleaned and polished are also used as drinking vessels. Powder horns were originally bovid horns fitted with lids and carrying straps, used to carry gunpowder. Antelope horns are used in traditional Chinese medicine. Horn may also be used as a material in tools, furniture and decorations, among other uses. A horn is valued for its hardness. A horn is somewhat thermoplastic and was formally used for many purposes where plastic would now be used. A horn may be used to make glue (Lundrigan, B. 1996).

Although no work on characterization of true horns has been reported, an experimental approach for evaluating the mechanical behaviour of hoof horn was developed by Dongsheng Zhang et al., 2007. The process is comprised of obtaining incremental slices of hoof horn, stamping samples from selected zones of the sectioned tissue, performing uniaxial tensile tests and evaluating the mechanical response using digital image correlation (DIC). In view of the limited experimental results on properties of this important natural composite material (cattle horn), the main objective of the present work is to study its hardness behaviour using nanoindentation technique and Vickers micro hardness tests.

2. EXPERIMENTAL PROCEDURE

Cattle horns were randomly selected and purchased from the Kano Abattoir. Five samples were selected and numbered A to E. the sample was prepared in order to obtain a well polished and etched sample for nanoindentation and Vickers micro hardness test. The nanoindentation test was performed using a nanoindenter (Micro Materials Ltd., Wrexham, United Kingdom) at the Ceramic Materials Laboratory, IIUM whereas Vickers micro hardness test was performed using Wilson Wolpert Vickers micro hardness machine. The five different samples are indented using diamond Berkovich indenter in sub microns and nano-scales. This device uses a pendulum pivoted on bearings which are essentially frictionless. The indenter is a three-sided pyramid, which is widely used for nanoindentation work because it can be machined down very accurately to a very sharp tip with a curvature radius of one half of a nanometer. The nanoindenter is capable of resolutions in the order of 0.1 µN and 0.1 nm for the load and displacement respectively. For the purpose of statistics and reliability, more than ten loading/unloading curves in each experiment were extracted and analyzed. A total of 15 indentation experiments for each specimen were made at a depth of 2 microns.

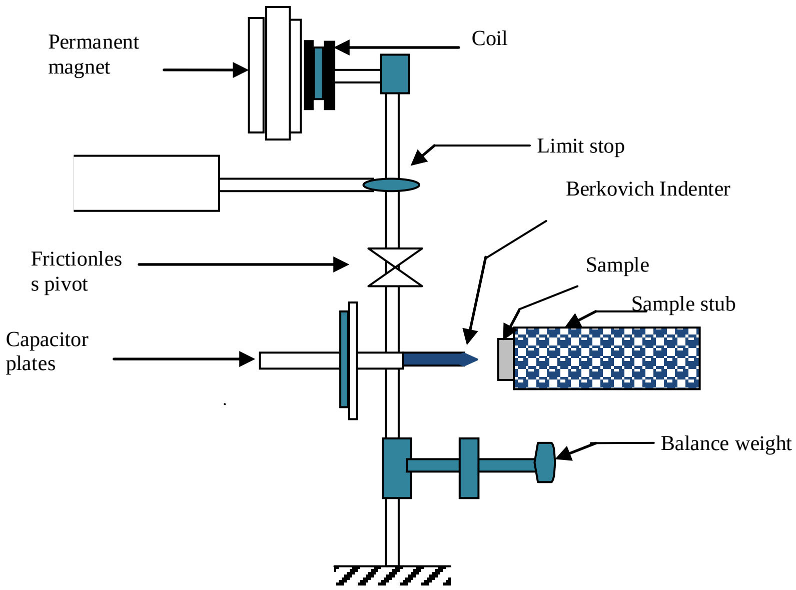


Figure 1: Experimental set-up for nanoindentation test

These two different types of hardness tests have effectively serve the purpose of investigating the hardness behaviour of the horn. The hardness tester controller, monitor and TV camera power was put on and the tester was set to zero for accuracy of the measurement. The work piece was placed on the vice and desired height was selected. The proper magnification was used to focus the image and the diamond indenter was changed to appropriate position. Finally, start button was pressed to measure the Vickers hardness for a waiting period of 20 sec. After the indentation, the magnifying lens turned back to center position for the dimension to be measured and confirmed by pushing the black button, the ocular was turned to 90^o (vertical position).

3. RESULTS AND DISCUSSION

Figure 2 shows the nanoindentation test loading and unloading curve for studying the mechanical behaviour of the cattle horn as a naturally produced thermoplastic material. The slope of the unloading curve provides a measure of elastic moduli [5]. The hardness was derived by dividing the load by the area of contact as mention in equation (1)

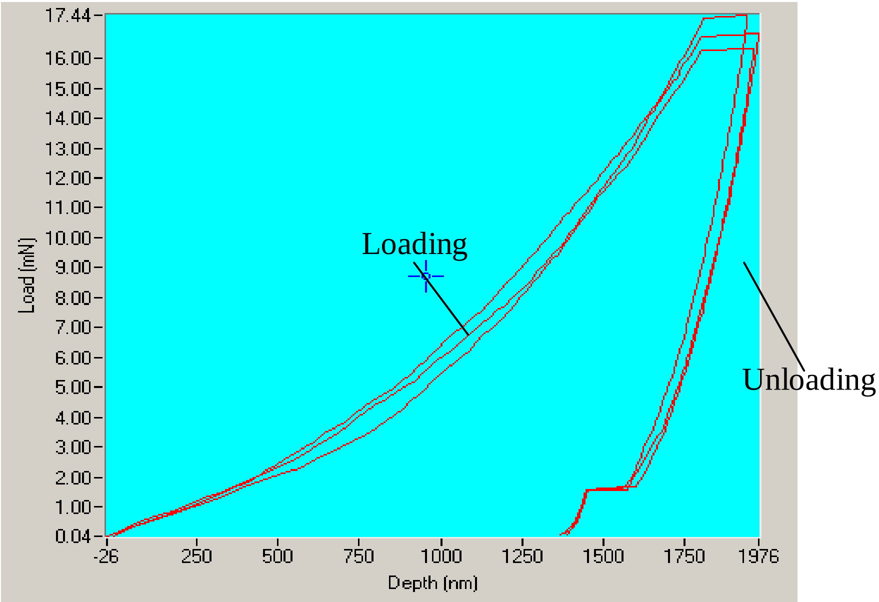
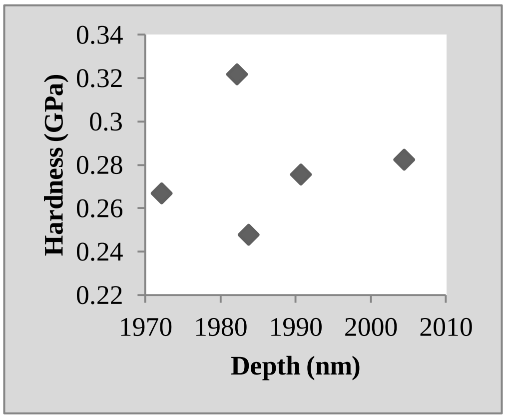
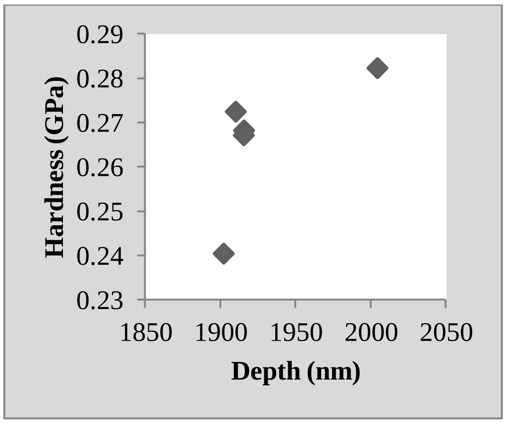


Figure 2: Loading and unloading curve for the cattle horn obtained using Berkovich indenter during loading and unloading

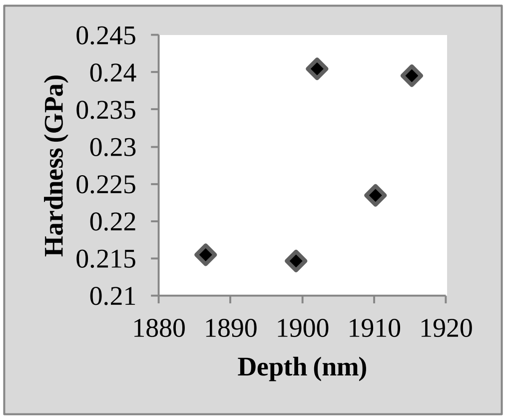
Figure 3 shows the hardness profile of five formulated cattle horn samples with respect to depth of penetration. It was observed that the value of hardness for all the samples increased with the increasing depth of penetration. This can be attributed to the fact that the value of hardness depends on the depth of penetration, h_c as mentioned in equation (1). The relation between hardness and the depth of penetration is inverse which shows that the indenter almost maintained the same contact area for all the samples during the test due to the hard nature of the true horn, which is in total compliance with nanoindentation theory, whereby hardness value is always dependent on load and contact area of the indenter tip. In this investigation a smaller and nearly same contact area was observed throughout the test with varying depth of penetration (as mentioned in equation 3) resulting in lower value of hardness which agreed well with the nanoindentation theory as stated earlier. However, it is noticed that hardness value increased with the increasing length of the horn (as shown in Fig. 3). This is due to the fact that the elemental composition of the horn was observed and found that it varies along the whole length (Abdullahi and Salihi, 2011). The range of hardness values of sample A, B, C, D and E obtained using nanoindentation results are 0.07-0.14, 0.22-0.27 GPa, GPa, 0.21-0.24 GPa, 0.24-0.28 GPa, and 0.24-0.32 GPa, respectively.



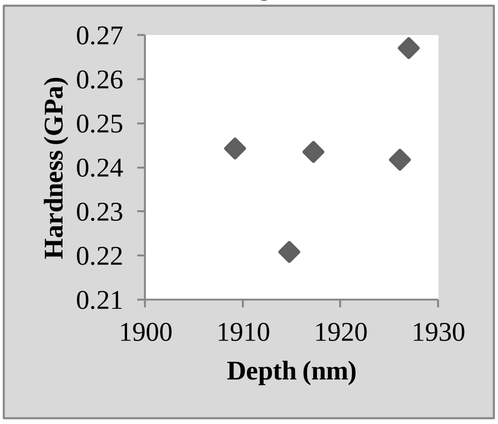
a



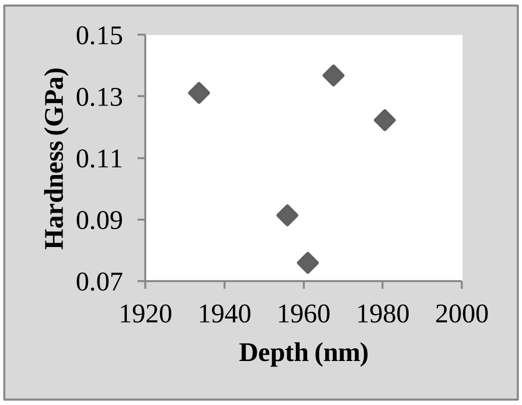
b



c



d



e

Figure 3: A graph of hardness against depth of penetration for the five cattle horn samples individually

Figure 4 present the same result of nanoindentation with all the five samples considered against the same depth of penetration at each point. The result also showed that hardness value increased with the increasing length of the horn until almost half of the lengths were the value increased dramatically and decreased slowly with the increasing length but increase and decreasing depth of penetration at the last two points respectively.

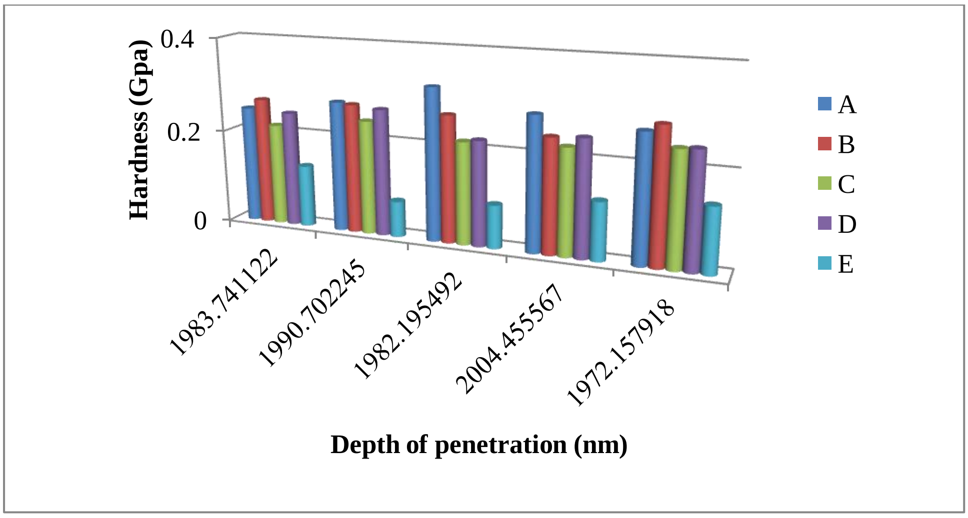


Figure 4: A graph of hardness against depth of penetration of five cattle horn samples

Figure 5 shows Vickers micro hardness results of the thermoplastic cattle horn material. In general, the hardness increases with the increase in length of the horn from the skull to the tip. Maximum hardness was obtained with the value of 26 HV for sample D and E. Similar trend was observed by Abdullahi and Salihi, 2011 in their study to characterize and investigate the potential application of the cattle horn in Kano as northern part of Nigeria. They employed tensile testing machine to investigate some of the mechanical properties such as tensile and bending to assess its suitability as one of the important engineering material. However, present study showed the maximum value of hardness for the cattle horn especially towards the middle of the length. The same authors also claimed that this cattle horn showed better tensile property, and the authors used different techniques to study the hardness behaviour as one of the mechanical property.

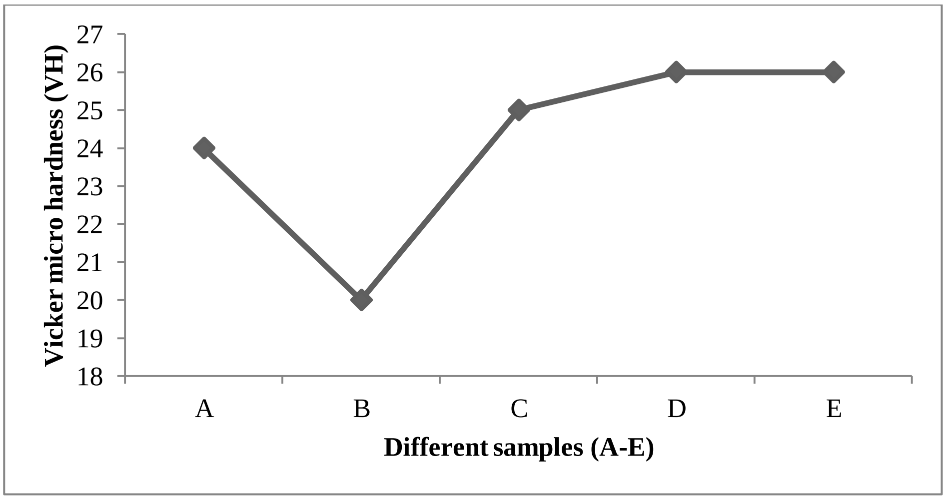


Figure 5: Variations of Vickers micro hardness with different samples

Figure 6 shows the profile of reduced modulus for the five formulated samples with respect to depth of penetration. It can be seen the value of reduced moduli of the cattle horn sample decreases with increasing depth of penetration to a certain depth and gradually increases as the depth increases, hence, it can be said that, the trend is really a zig-zag shape showing the hardness behaviour and the elastic deformation formed by the indenter. From the present experimental results it is also observed that reduced moduli increases with the increase in hardness of the material. The increase in reduce moduli indicate the hardness of the material that depends on the area of contact and the ductility of the material. For any hard material such as silicon and steel and using the same indenter the expected result is alike in following the same trend [5] of load displacement curve.

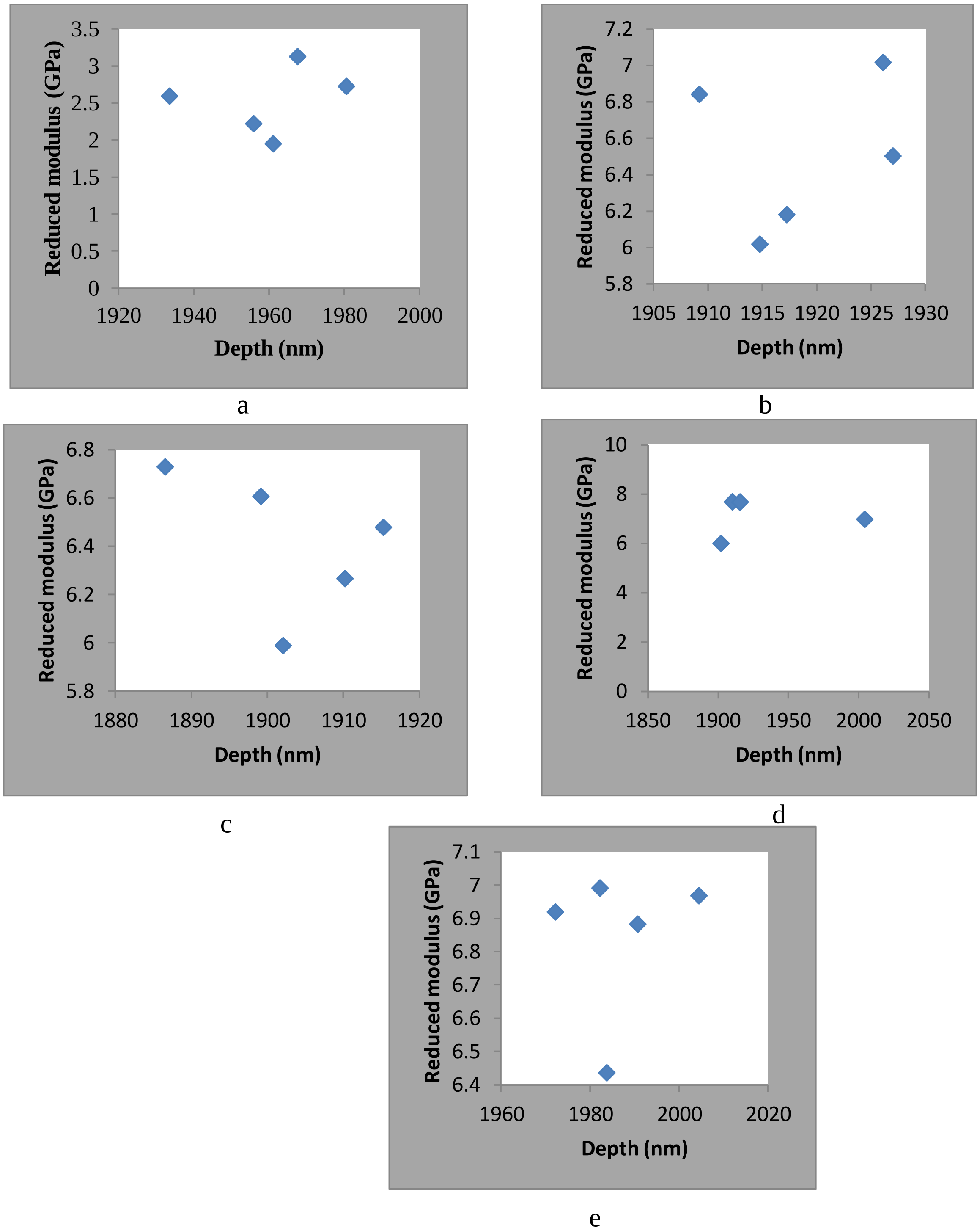


Figure 6: Reduced modulus against depth of penetration for the five cattle horn samples individually

4. CONCLUSION

The results of micro and nanohardness test showed that the value of hardness is increasing with the increasing length of the horn. The hardness increase from to the skull to the middle of the horn length and starts decreasing gradually to the tip of the horn. Both tests confirmed that the hardness value is approximately higher at the middle of the horn.

5. ACKNOWLEDGEMENT

Authors are grateful to the Department of Manufacturing & Materials Engineering, International Islamic University (IIUM) which made this study possible**.**

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