

EVALUATION OF MICROHARDNESS OF A BRAND OF STAINLESS STEEL K FILES

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ABSTRACT

The objective of this study was to determine and compare the microhardness values of a brand of stainless steel K files (Mani, Inc. Japan), acquired from Pakistan and United Kingdom, in accordance with AS 1411. This study was conducted at the Institute of Space Technology, Islamabad, over a period of two months. A total of 40 stainless-steel K files (Mani, Inc. Japan) of identical size (ISO#25), were collected and divided into two groups. Group A consisted of 20 K files purchased from the Rawalpindi/ Islamabad city in Pakistan, while Group B consisted of 20 K files that were purchased from London, United Kingdom. The microhardness of files belonging to both groups was determined using Vickers microhardness tester. The data were statistically analyzed using independent sample t test. No significant differences between microhardness values of Group A and B were found. However, substantial variations within each group were observed. Variations found between the identical file sizes of the same brand suggest that the manufacturing processes involved in the fabrication of endodontic files may not be prudently controlled.

Key Words: *Stainless steel K files, microhardness, strain hardening.*

INTRODUCTION

Hardness is not an inherent physical property but an attribute of a material.¹ It is defined as resistance of solid matter to a permanent change in shape due to application of compressive forces.² Reliant on the amount and type of force applied, solids usually have three responses. They show elastic behavior, which is the capacity to change form temporarily but return to the original form upon removal of the applied force. Plasticity is the permanent change in form but without fracture, in response to the applied force, and fracture when the force exceeds the ultimate tensile strength of the material.²

Over the decades, several test systems for gauging the hardness of materials have been introduced, such as scratch tests and indentation tests. The simplest form of bar scratch testing was introduced as early as 1722.³ Reforms in these basic testing systems took place over the years and in 1822, hardness testing system, currently known as Mohs scale was introduced that was based on scraping material surfaces with a diamond and quantifying the breadth of the resultant line.³ In 1859, earliest forms of static indentation tests were

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Received for Publication: October 3, 2017
Revised: December 20, 2017
Approved: December 21, 2017

introduced.⁴ Indentation tests are generally based on the formation of indentation on the surface of a metal or ceramic and hardness is established by gauging the perpetual depth of the indentation and the applied load.⁵ Generally, the indentation hardness may be described in terms of plastic and, to a lesser extent, the elastic properties of the metal or ceramic concerned. When applying a static force (load) with a particular indenter, smaller the indentation measured, harder the material.⁶

JA Brinell in 1900 proposed the first largely recognized and standardized indentation-hardness test known as the Brinell hardness test. This test employs a load of up to 3,000 kilograms to indent the metal surface using a steel, tungsten carbide or diamond spherical indenter, made of 1 to 10 millimeter diameter. Hardness is gauged using an optical microscope by measuring the average of two readings of the indentation diameters at right angles.⁶

In 1924, Smith and Sandland introduced the Vickers hardness test method, as an alternative to the Brinell hardness test. The Vickers hardness test procedure is commonly employed for small sections or thin components and is based on an optical measurement system.⁶ The micro hardness test method, ASTM E-384, recognizes a variety of light loads employing a square based pyramid shaped diamond indenter to form an indentation that is measured and mathematically converted into a hardness value using the formula,²

Psin (θ/2)/L2.⁷ Normally, loads range from a few grams to one or several kilograms. This method can be used for testing metals, ceramics as well as composites.⁷

Later, in 1939, Fredrick Knoop introduced an alternative to the micro Vickers hardness test, signified as the Knoop hardness test. This hardness test utilizes a shallower and more extended form of the diamond indenter. This test procedure permits more precise hardness measurement of brittle or thin materials, since it is intended for use under test loads lower than the Vickers hardness test.⁶ Currently, both these test procedures continue as common hardness investigation methods.

The Australian standard for cutting tools, AS 1411, specifies a hardness range of 550-650 VHN.^{8,9} Several researchers have contributed towards evaluating the microhardness values of endodontic files in the past. For example, in 1962, the Knoop hardness values for stainless steel files reported by Craig and Peyton ranged from 525-565.¹⁰ In 1996, Brockhurst and Denholm reported significant differences in microhardness between endodontic files from two different manufacturers.⁸ In their study, the Vickers hardness range was from 400 to 651 VHN, falling below the hardness required for cutting instruments (550-650 VHN). In 1998, Brockhurst and Hsu reported values ranging from 522-542 VHN for stainless steel endodontic files.¹¹ In 2004, Darabara, M et al, reported significant differences in the Vickers micro hardness values for 3 different stainless steel endodontic file brands.¹² They suggested that the differences in hardness are independent of the alloys used and should be accredited to the thermo mechanical treatment of alloys during manufacture.

Information about the surface hardness of an instrument can be used as a guide to its ultimate tensile strength and its cutting efficiency.¹³ ISO 3630-1 for K type files and reamers only specify that the working part of the instrument be made of either stainless steel or carbon steel, with only minimum requirements set for the material used.¹⁴ The type of steel and its treatment shall be at the discretion of the manufacturer. Owing to the dearth of information regarding the surface hardness properties of endodontic files, this study was aimed at evaluating and comparing the microhardness of a brand of stainless K files (Mani Inc. Japan),

acquired from Pakistan and United Kingdom. Files from one particular brand were selected because of the availability of non-standardized and poorly machined files of this brand in the local and international markets.¹⁵ Reported discrepancies in their geometry and surface topographical features call attention towards evaluation of the mechanical properties of the files.¹⁵

METHODOLOGY

The description about the files used for the testing purpose in this study is given in Table 1. In order to compare the microhardness values of the two groups of files, ZHV30-A Zwick Roell low load Vickers microhardness tester was used (Indentec Hardness Testing Machines Limited, West Midlands United Kingdom). Testing was done in accordance with ASTM E-384.⁷ Samples were prepared by separating the handles of the files and mounting them on bakelite using Metkon, Metapress-M mounting press (Metlab Corporation, Hyde Park, New York). After retrieving the mounted instruments from the press, the samples were subjected to grinding and polishing using Metkon Forcipol 2V (Metkon Instruments Inc. Turkey) to obtain a flat surface. Some mounted samples after final grinding and polishing are shown in Fig 1.

The indentation of samples was done by pyramid shaped diamond indenter; employing a static force application technique. A load of 300 g for 10 seconds was applied perpendicularly onto each specimen.⁷ Each file was indented at three points, first indent being made at the center which was designated as point B, and the next two indents made towards the tip and the edge, designated as points A and C respectively, having a minimum of three indentation spaces between each indent.⁷ An average microhardness value for each file was then derived.

RESULTS

The lowest value recorded for Group A was 478 HV and the highest was 601 HV. Similarly the lowest value obtained for Group B was 499 HV as compared to the highest recorded value of 618 HV. The acquired data was statistically analyzed using independent sample t test. The average of microhardness values calculated for each sample in Groups A and B are given in Table 2.

TABLE 1: FILES USED FOR TESTING

Groups assigned	Area of purchase	File specification	No. of files	Manufacturer	Lot number
Group A	Pakistan	Stainless steel K files 21mm # 25	20	MANI, INC. 8-3 Kiyohara Industrial Park. Utsunomiya, Tochigi. Japan	R151412100
Group B	United Kingdom	Stainless steel K files 21mm # 25	20	MANI, INC. 8-3 Kiyohara Industrial Park. Utsunomiya, Tochigi. Japan	R110868200

TABLE 2: AVERAGES OF MICROHARDNESS VALUES OF GROUPS A AND B

Sample number	Group A	Group B
1.	505 HV	499 HV
2.	543 HV	554 HV
3.	499 HV	564 HV
4.	478 HV	547 HV
5.	561 HV	586 HV
6.	502 HV	564 HV
7.	550 HV	554 HV
8.	590 HV	547 HV
9.	561 HV	583 HV
10.	594 HV	527 HV
11.	575 HV	590 HV
12.	568 HV	564 HV
13.	599 HV	568 HV
14.	548 HV	579 HV
15.	566 HV	618 HV
16.	592 HV	549 HV
17.	581 HV	523 HV
18.	601 HV	536 HV
19.	564 HV	558 HV
20.	491 HV	554 HV

TABLE 3: MEANS AND STANDARD DEVIATIONS OF MICROHARDNESS VALUES OF GROUP A AND B

Groups	N= 40	Mean of averages	P value
A	20	553.4 ±38.6	0.6
B	20	558.2 ±26.4	0.6



Fig 1: Mounted specimens for microhardness testing

Statistical analysis by independent sample t test showed no significant differences ($p>0.05$) between the two groups. Means and standard deviations of both the groups are summarized in Table 3.

DISCUSSION

The microhardness results observed in this study were found in accordance to previous studies.¹² No significant differences between the two groups were found. However, substantial variations in the hardness values between the instruments tested were observed in our study. The higher values of microhardness seem suitable, since they fall within AS 1411 (550-650 VHN).⁹ On the other hand, the lower range values of microhardness suggest that there is a potential for substantial improvement to the strength of these instruments.⁹

Higher microhardness value is generally attributed to two strengthening mechanisms. Predominantly because of work hardening imposed by the plastic deformation developed during cold drawing process and secondly grain boundary strengthening (Hall-Petch effect) material.¹ The extent of a metal's plastic deformation indicates towards its strength and hardness. Plastic deformation occurs through slip mechanism provided by line defects called dislocations.⁴ The movement of dislocations along the slip planes helps in deformation of a metal at a relatively lower applied stress. In order to increase the hardness of a metal, one way is to restrict the movement of dislocations by their interaction with each other and interstitial atoms. When a metal is deformed, the dislocation density increases.⁴ High interaction among the increasing number of dislocations creates hindrance in their movement.^{16,17} Eventually, the increased amount of entanglement among the dislocations results in an increase in the yield and tensile strength of a metal.¹⁶

Excessive cold working however, may result in an increase in the dislocation density of a metal to as high as 1012 cm square, thus significantly reducing ductility and increasing the likelihood of a brittle failure.¹⁶ This happens because as dislocation density increases, due to applied stresses above the yield point, it becomes increasingly difficult for the dislocations to move because their strain fields interact with each other. A material that already has a high dislocation density can only deform but so much before it fractures in a brittle manner.¹⁶

Another method of strengthening materials, basically by altering the grain size is called as grain-boundary strengthening (or Hall-Petch strengthening).¹ Reduction in the grain size results in an increase in the hardness and strength of a metal. It is based on the fact that the increasing number of grain boundaries hinders dislocation movement and increases the yield strength. Grain size can be altered by heat treatment after plastic deformation and altering the rate of solidification.¹

Prior studies have revealed that manufacturing procedures are related with the fracture dynamics of endodontic files 1. Inapt surface hardness may render

the instruments either brittle or they may have a reduced cutting efficiency.¹⁸ In annealed state, the hardness of austenitic stainless steel is around 300 HV.¹² Therefore, it has a higher resistance to rupturing in the annealed state but can become highly susceptible when heavily cold worked.¹⁸ Maximum hardness values however, are essential to conserve the sharpness of cutting edges and to prevent wear and breakage of the instruments.¹⁸ Selection of appropriate materials for endodontic instruments requires a balance between strength and ductility. These two variables are interconnected, in that higher the strength, lower the ductility. The top quality materials will display a paramount amalgamation of these two attributes.¹⁸

Variations found between the identical file sizes of the same brand in our study advocates that the manufacturing processes involved in the fabrication of endodontic files may not be prudently controlled.⁸ One must bear in mind that hardness is not an intrinsic physical property but rather depends upon the treatment and microstructure of a material.³ Therefore, cold working and heat treatments are significant fabrication techniques which need careful monitoring during file fabrication for optimum surface hardness properties.¹⁸ It should however be considered that within the limitations of our study, only a single brand of files was tested for evaluation of surface hardness. Further research with several brands of files in multiple sizes is required to acquire a more definitive conclusion on this subject.

CONCLUSIONS

Within the limitations of this study, there were no significant differences between the locally and internationally acquired files, manufactured by the same company. However, substantial variations of microhardness values was found within each group. Manufacturing techniques, particularly strain hardening of the instruments need further evaluation to better understand the cause for the variations in microhardness identified in this study.

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CONTRIBUTIONS BY AUTHORS

- 1 Maryam Saeedullah:** The main idea in the study belongs to Author 1. She designed the study models and conducted the practical tests for data collection. Moreover, she has written the article.
- 2 Syed Wilayat Husain:** Helped in arranging all the necessary equipment required for the testing purpose. He supervised the projects and contributed towards devising established methodological approaches for the study. He also helped with the statistical analysis.