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PHYSICO-MECHANICAL PROPERTIES OF IRON-MILLSCALE REINFORCED CERAMIC COMPOSITE

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ABSTRACT

The overall safety of an aircraft requires that structural parts meet operational aerodynamic critical mechanical characteristics. This paper evaluated two critical mechanical properties of iron millscale (IMS) particles reinforced ceramic matrix composite (CMC) as a suitable material for aircraft structural application. The IMS particles reinforced CMC was produced by powder metallurgy method. IMS particles addition varied from 5 – 30 wt. % in a matrix comprising a mixture of silica, and magnesia and sodium bentonite as lubricant. The composites were subjected to physical and mechanical properties tests while their microstructures were characterised using scanning electron microscopy coupled with energy dispersive spectroscopy (SEM/EDS) and the data obtained analysed. The results show promising performances by the composites in terms of a relatively low density (2.75 g/cm^3) as a precursor of lightweight, enhanced specific strength of 58.3 kN.m/kg and desirable impact toughness up to 77.8 J. Resistance to lateral reduction in dimension as indicated by the compressive strength is 168.2 MPa. It is concluded that the intrinsic elastic transformation behaviour of the reinforcing phase during sintering is responsible for the mechanical properties enhancement.

Keywords: C*eramic matrix composite, iron millscale, specific strength, impact energy*

INTRODUCTION

The quest for sustainable safety and operational efficiency in the aerospace industry cannot be divorced from the design and manufacture of aircrafts with materials exhibiting exceptional functional characteristics. Component performance in this industry is primarily determined by mechanical properties such as strength, toughness as well as physical property such as density in consideration of weight. Despite a great deal of research efforts that have been expended in this direction, the reality today still suggests that meeting the goals of

sustainable safety and efficiency in the aviation industry remain work- in-progress. This submission is sequel to the challenges of developing advanced materials that can effectively replace the traditional super- alloys such as Nickel-Titanium alloys and 7075 Aluminium alloys (Peters and Leyens, 2003).

According to Standridge (2014), the need to develop materials of high specific strength coupled with significant impact toughness for safety and minimal casualty in the case of unavoidable crash is imperative.Ceramic matrix composites (CMCs) have been identified as one of the promising advanced composite materials with capabilities for exceptional performance (Nageswara, 2011). It is also generally believed that the use of CMCs in advanced engines (turbine) can be safely extended to comparable engines that operate in high temperature environments (Randelovic*et al.* 2012). The most sought after functional characteristics of aircraft structural components such as nose-cap, fuselage, wing, etc., include excellent thermal shock resistance, high mechanical strength (shear strength, impart toughness, flexural strength and hardness) and a relatively low weight. These properties can be induced in the composite through innovative materials processing and engineering improvements.

It is envisaged that the use of such composite will boost the chances of surviving harsh thermo-mechanical environments that aircrafts are known to operate.This underscores the use of a combination of materials as matrix in developing certain category of composites in order to effectively harness property synergy which such materials may offer. The development of hybrid composites is a typical example of such synergy. However, the production of good quality CMCs cannot be divorced from the constituent materials particularly in terms of the type, nature, and condition of the reinforcement phase. The works of Lee and Speyer, 2002, Zhu *et al.* (2007), Chmielewski*et al.* (2012), Tarabay*et al.* (2013) and Nagesh*et al.* (2014) established a profound influence of homogeneous dispersion of fine reinforcement particles on both physical and mechanical properties of the various ceramic matrix composites developed. According to Riina*et al.* (2014), iron-based sintered friction materials are best suited for structural application where temperature during service may go up to 1100^0C. Whereas, copper-based sintered friction materials can only withstand up to 600^0C. Further, iron-based sintered friction materials are less costly compared to the latter (Hicks, 2006). This paper evaluates the specific strength and fracture toughness of iron millscale (IMS) reinforced silica-magnesia matrix composite with a view of determining its suitability as aircraft structural material.

MATERIALS AND METHOD

Materials

The reinforcement material used in this study is iron millscale particles (IMS) while the matrix is a mixture of two ceramic materials namely, silica and magnesia. Iron millscale particles were sourced from Universal Steels, Lagos, Nigeria. Silica sand was obtained from the beach of the Lagos Atlantic Ocean (bar beach). Magnesia and bentonite powders were obtained from a local chemical vendor but were manufactured in China and Wyoming, USA respectively.

Methods

The materials composition was determined using X-ray fluorescence (XRF) spectrometer (model, ARL9400XP+ Thermo, Switzerland) and the results are shown in Table 1(a-c).

Table 1a: Chemical composition of iron millscale

Compound	FeO	Fe_2O_3	Fe_3O_4	SiO_2	MgO	CaO	MnO	L.O.I
Wt. %	68.809	24.735	6.184	0.011	0.016	0.220	0.024	0.001

Table 1b: Chemical composition of silica sand

Compound	SiO_2	Al_2O_3	Fe_2O_3	FeO	CaO	MnO	MgO	Na_2O	K_2O	M.O.I	L.O.I.
Wt. %	98.988	0.180	0.152	0.024	0.016	0.009	0.024	0.003	0.002	0.001	0.491

Table 1c: Chemical composition of magnesia

Compounds	MgO	CaO	SiO_2	M.O.I	L.O.I
Wt. %	98.840	0.025	0.007	0.001	0.012

Note: M. O. I. = Moisture on ignition and L. O. I. = Loss on ignition.

Materials Milling and Blending

The as-received iron millscale was milled in a ball mill and sieved to 106 µm using a standard BSS sieve. Silica sand was also milled and sieved to 212µm. Optimised quantity of silica sand particles (10 wt. %), magnesia powder (25 wt. %), and bentonite clay powder (65 wt. %) were used as matrix. The iron millscale particles at varied wt. % of 0 % (control), 5 %, 10 %, 15 %, 20 %, 25 % and 30 % were separately mixed with 100 %, 95 %, 90 %, 85 %, 80 %, 75 %, and 70 % by weight of the matrix blend respectively. A uniform distribution of reinforcement particles in the matrix was achieved by mixing the powders prepared for each sample group in the mixer for 20 minutes at 50 rpm. Distilled water in the

proportion of 12 % by weight of the total mixture was added and the mixture was manually mixed again.

Composite production

The particulate ceramic matrix composites were produced by powder metallurgy technique which encompasses compacting and sintering of the particles to achieve a significant solid state diffusion of the grains and good vitrification of the particles. The wet-blended samples were fed into metal moulds of desired shape and green samples were obtained by cold uniaxial pressing using a hydraulic press at 30 MPa. The samples were dried in open air for 3 days, followed by drying in an oven at 110^0C for 24 hours to expel any moisture left in the composite and to avoid crack during sintering.

To enhance bonding of the compacted particles, the samples were gradually heated to a temperature from 500^0C - 800^0C which is below the melting point of the constituent materials but high enough to develop significant solid state diffusion. Thereafter, sintering of the samples was carried out in a heat treatment furnace pre-set at heating rate of 10^0C/min in the following sequence: (i) Heating to 700^0C and allowed to soak for three hours, (ii) Heating to 1000^0C and allowed to soak for three hours, (iii) Heating to 1200^0C and allowed to soak for three hours, and (iv) finally heating to 1350^0C and allowed to soak for three hours. The samples were then removed from the furnace and allowed to cool after which they were characterised.

Properties Evaluation

Microstructure

The microstructural features of the test samples were examined using a scanning electron microscope (model, ASPEX 3020) equipped with Noran-Voyager energy dispersive X-ray spectroscope (EDS). The samples were mounted on a conductive carbon imprint left by the adhesive tape prepared by placing the samples on the circular holder and coated for five minutes to enhance their electrical conductivity. The samples were then analysed at an accelerated voltage of 15 kV for the energy dispersive X-ray spectroscopy.

Density

Weights of the sintered samples in air were measured with an analytical balance whereas their weights in water were measured with a suspension kit and measuring cylinder at room temperature. The densities of the sintered samples were then determined using equation 1.

$$\text{Density } (\rho) = \frac{\text{Mass (g)}}{\text{Volume (cm}^3\text{)}} \quad (1)$$

Hardness test

Hardness test was conducted on the samples in accordance with ASTM E10 standard using a Brinell hardness measuring machine of 20 kN capacity. The sample was mounted on the machineand a load of 5 kN was applied on it for about 10 seconds. Then, the diameter of indentation left in the sample surface was measured with a low powered microscope. To eliminate possible segregation effect, a minimum of three hardness readings were taken for each sample at different locations.The samples'Brinell hardness values werecomputed using equation 2.

$$\text{Hardness (HBN)} = \frac{2P}{\pi D[D-\sqrt{D^2-d^2}]} \quad (2)$$

Where:
P is load (kgf)
D is diameter of indenter (mm)
d is the diameter of indentation (mm)
$\pi = 3.142$

Compressive strength test

The samples used for the compressive strength test were prepared in accordance with ASTM D695 standard. With the aid of an Instron universal testing machine (model 3369K, USA) each of the samples was subjected to uniaxial compressive loading until the sample breaks and the strength value recorded. The compressive strength of a material is its capacity to withstand loads tending to reduce its size. On an atomic level, the molecules/atoms are forced apart when in tension whereas in compression, they are forced together. Since atoms in solids always try to find an equilibrium position/distance between each other, stresses arise throughout the entire material which opposes both tension and compression.

Impact energy test

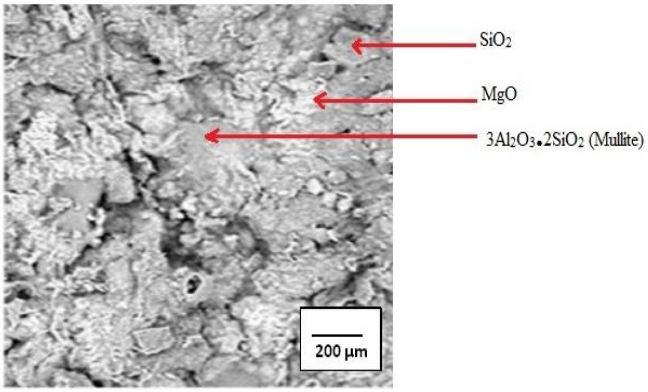
The samples were prepared in accordance with ASTM D790 standard with each sample having a 2mm deep V-notch at the center. Each sample was clamped vertically with the notch facing the striker and the striker swings downwards impacting the sample at the bottom of each swing cycle. Each sample was subjected to horizontal impact loading using an Avery impact testerunder a striking pendulum velocity of $5ms^{-1}$from a height of 1.3 m. The energy absorbed to fracture each specimen was read-off the instrument'sdynamometer.

RESULTS AND DISCUSSION

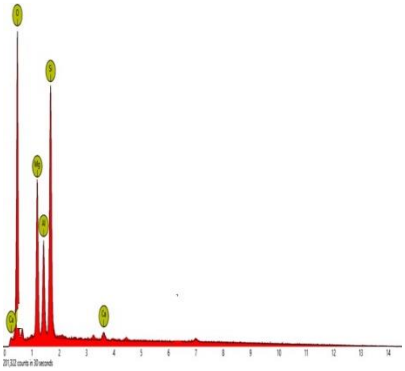
Microstructure

Scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS) tools were used in examining the microstructure of the ceramic matrix composites. The SEM micrographs and EDS spectrographs are shown in Plates 1-3. The micrographs show the composites' heterogeneous make-up in terms of different mineralogical phases present and varied reinforcement particles morphology which are globular and needle-like. The EDS spectrographs show the presence of O, Si, Al, Mg, Fe, and Ca but the unreinforced (control) sample contains no Fe. There are also indistinguishable peaks in the spectrographs indicating the presence of other elements in trace amounts.

Generally, the white spots in the micrographs are magnesia particles while the dark spots are FeO particles from the iron millscale. The ash colour and needle-like region of the matrix indicates the presence of mullite ($3Al_2O_3 \cdot 2SiO_2$), a highly crystalline compound from bentonite clay-silica mixture formed at 1350^0C sintering temperature. As evident in the micrographs shown in Plates 2 - 4, the iron millscale (FeO) particles are homogeneously distributed within the matrix without any form of segregation. The interfacial bonding between the reinforcement particles and the matrix is enhanced by the effective lubrication provided by the mullite clay (bentonite) resulting in a good vitrification during sintering

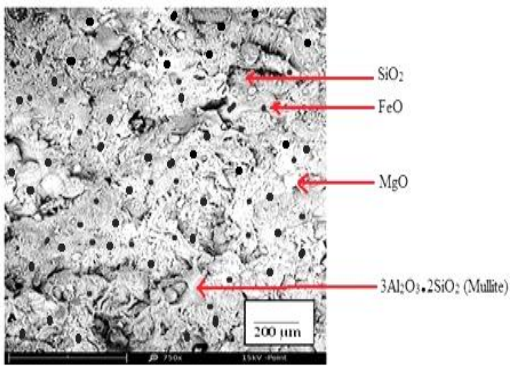


(a)



(b)

Plate 1: (a) SEM and (b) EDS of ceramic matrix composite without iron millscale addition



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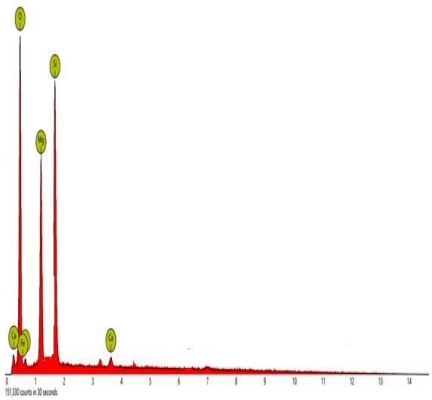
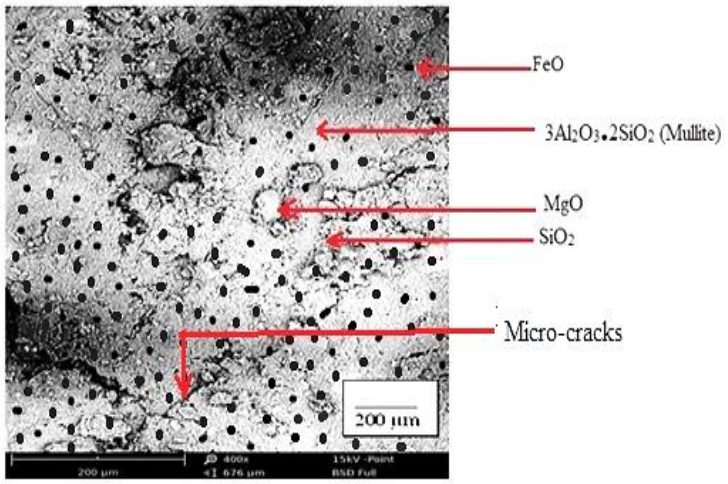


(b)

Plate 2: (a) SEM (b) EDS of 10 wt. % iron mill scale reinforced ceramic matrix composite



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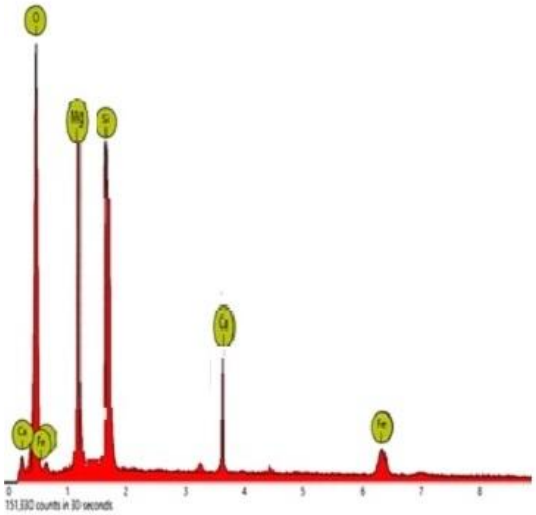

(b)

Plate 3: (a) SEM (b) EDS of 30 wt. % iron mill scale reinforced ceramic matrix composite

Physical and Mechanical Properties of IMS Reinforced Ceramic Matrix Composite

Table 2 shows the results of physical and mechanical tests carried out on the ceramic matrix composites at varied iron millscale (IMS) additions. The results are illustrated in Figures 1-2.

Table 2: Physical and Mechanical Properties data of ceramic matrix composites at varied IMS addition

Wt. % IMS	Density ($g.cm^{-3}$)	Hardness (HBN)	Compressive strength (MPa)	Impact energy (J)	Specific strength ($kN.mkg^{-1}$)
0	2.31	190	126.13	40.23	54.60
5	2.42	175	137.44	52.41	56.79
10	2.51	162	143.41	57.27	57.08
15	2.57	153	148.57	65.03	57.91
20	2.75	146	160.18	77.81	58.25
25	2.98	132	168.17	89.07	56.42
30	3.07	127	164.13	85.18	53.51

Density

From the properties data in Table 2, the unreinforced ceramic matrix composite (CMC) has a density of 2.31 g/cm^3 while the IMS reinforced CMC at 30 wt. % developed a density of 3.07 g/cm^3.There is a progressive increase in the densities of the reinforced CMCs as more IMS particles were added. This may be attributed to a relatively high packing factor of millscale particles as its concentration within the matrix increases thereby increasing the density of the composites. Due to the intrinsic density of iron, it is established (Asif*et al*., 2011) that iron based friction materials has the tendency to develop high density. However, a relatively low density that confers lightweight regime for energy efficiency is preferred in materials that are employed in advanced devices. Thus, the 2.75 g/cm^3 density induced in the composite at 20 wt. % compared favourably with similar lightweight materials with enhanced specific strength and toughness. For example, the asbestos based material used in the production of a civilian airbus brake pad has a density of 1.89 g/cm^3 which translates to a difference of 0.86 g/cm^3compared with the density of IMS reinforced CMCs produced in this study.

Compressive Strength

The compressive strength of a material is the measure of its capacity to withstand loads tending to cause lateral reduction. As evident from Table 2, the composites compressive strength increases with increasing wt. % of IMS particles addition. However, the unreinforced composite exhibited the lowest compressive strength of 126.13 MPa while the highest compressive strength of 168.17MPawas achieved at 25 wt. % IMS. The 106 µm fine particles enhanced grain densification giving rise to improved compressive strength. According to Lee *et al.* (2002), fine particles are known to enhance strength than coarse particles of the same weight fraction because smaller grain sizes increase the frequency with which

dislocations encounter grain boundaries, thus requiring larger stresses for deformation to occur. Comparing the compressive strength value of 168.17MPa with the results of similar previous works on existing conventional advanced structural materials shows that the developed composite has a relatively higher compressive strength. This is a clear indication that the developed composite will compete favourably with existing conventional advanced structural materials.

Specific strength/Impact energy

It is required that materials meant for application as advanced structural components should exhibit reasonably high capacity against stress combined with light weight that ensure safety and comfort. This property is referred to as specific strength, usually evaluated through the strength-to-density ratio or simply strength/weight ratio of the material. In this study, the composites' (CMC) specific strength is obtained by dividing the compressive strength values at varied iron millscale addition with the density developed in the composite and the values obtained are illustrated in Fig. 1. The Figure shows a progressive marginal increase in specific strength as the reinforcement increased from 5 - 20 wt. %, then decreased sharply monotonously due to high density. For an enhanced specific strength, a huge weight reduction is required. This can only be achieved through innovative processing such that the material can still exhibit desirable structural integrity after the attainment of lightweight without compromising other parameters such as low shrinkage, tenacity and good toughness. At relatively low densities between 2.42 g/cm^3 and 2.75 g/cm^3, the specific strength developed in the composite range from 56.8 – 58.3 $kN.mkg^{-1}$.These values compared well with values obtained in composite airframe stress analysis (Muniyasamy and Moorthy, 2012). Generally, specific strength property of a material is of fundamental importance in consideration for aerospace and automobile applications.

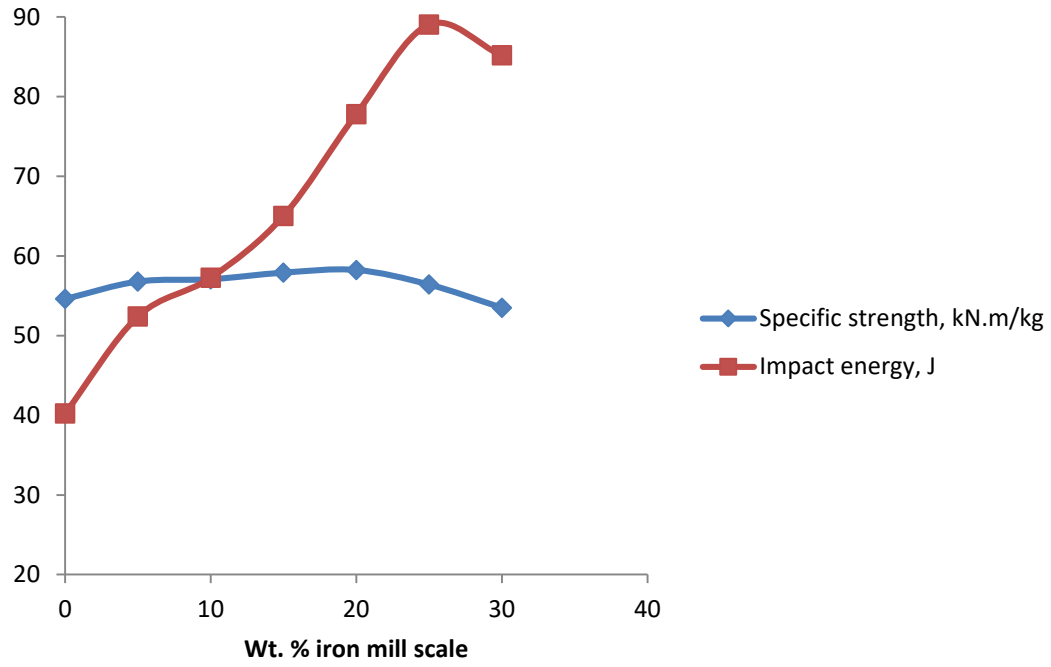

Fig. 1: CMC specific strength/Impact energy variations with iron millscale addition

For the purpose of virtual comparison, the impact energy result was plotted together with the specific strength. The impact energy is an indication of the level of toughness exhibited by the composite which is observed to increase progressively from 52.3 – 89.1 J as the IMS reinforcement particles increased from 5 – 25 wt. %. The concurrent increase in impact energy with increase in weight fraction of IMS particles must have conferred on the ceramic composites an enormous capacity to absorb energy. Invariably, increasing the weight fraction of iron millscale particles enhanced the composite ceramic matrix elastic property thereby increasing its toughness in agreement with Kaundal *et al.* (2012). However, as the weight percent of the reinforcement increases beyond 25 wt. %, micro-cracks develop (see Plate 3a) resulting in impact energy reduction from 89.1 J to 85.2 J. This may be attributed to poor interfacial adhesion between the ceramic matrix and the reinforcement particles. The rather unmitigated decrease in impact strength beyond 25 wt. % of millscale particles addition is due to the inability of the reinforcements to block the crack developed from propagating. This assertion compares well with Thomas *et al.* (2012) that the inability of metal

particles to disperse the energy of propagating cracks will give rise to decrease in impact energy or fracture toughness of the material. The mechanism by which ceramics prevent micro cracks propagation entails tetragonal phase transformation to monoclinic which closes the crack and stops it from propagating. This phenomenon is referred to as increase of tenacity by phase transformation that occurs during sintering. On the basis of lightweight, high strength coupled with good impact energy, it is evident from Fig. 1 that the CMC demonstrated a balanced specific strength (56.8 -58.3 kN.m/kg) and impact energy (52.4 – 77.8 J) between 5 wt. % and 20 wt. % IMS additions. These properties values compared well with existing conventional materials.

Hardness

As presented in Table 2 and illustrated in Figure 2, the unreinforced (0 wt. %) sample exhibited the highest hardness value of 190 HBN. This is attributable to the relatively large presence of silica (SiO_2) and alumina (Al_2O_3) that make-up the matrix. According to literature (Hassan*et al.*, 2010, Siddique and Khan, 2011), silica and magnesia are known to be among the hardest substances. In this study, the developed composites demonstrated the highest hardness value of 175 HBN at 5 wt. % iron millscale addition. The particle size effect of metallic phase particles according to Sbaizero*et al.* (2000) often modifies composites hardness behaviour. Thus, the fine 106 µm iron millscale particles must have enhanced the composite densification in agreement with Correa *et al.* (2004) and Guazzato*et al.* (2004) that fine grains enhance densification which correspondingly improves the mechanical properties of composites. However, as shown in Fig. 5, there is a progressive decrease in the hardness values as the weight fraction of the iron millscale particles increased. This is due to increase in the ductile metal phase (FeO) compared to the hard ceramic matrix phase being a mixture of silica and magnesia.

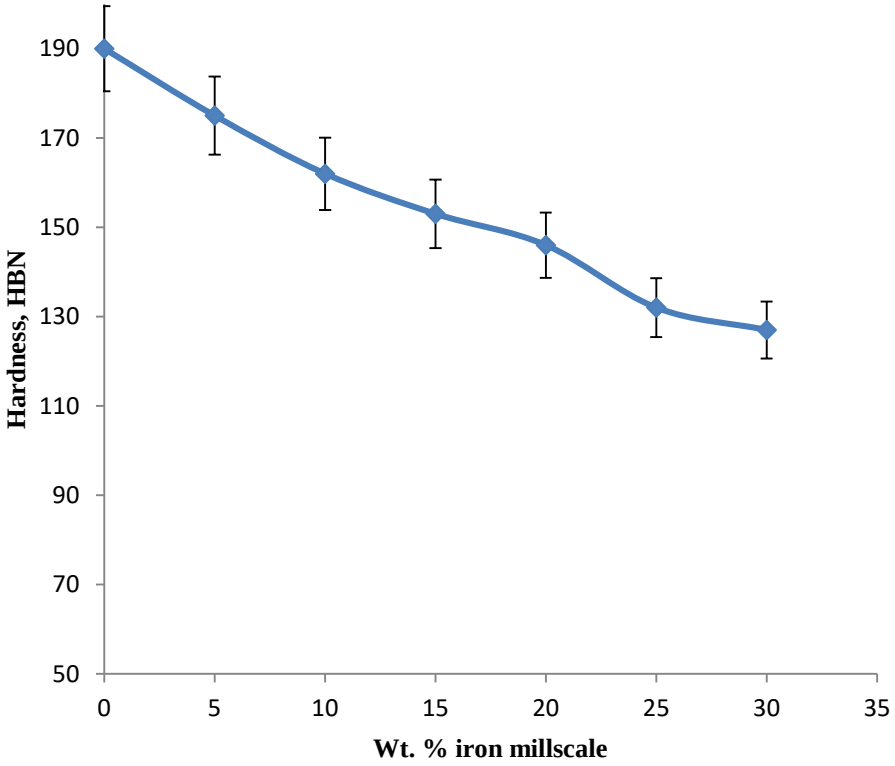

Fig. 2: CMC hardness variation with iron millscale addition

CONCLUSION

The evaluation of the specific strength, impact energy and other selected relevant physical (density) and mechanical (compressive and hardness) properties of ceramic matrix composite (CMC) reinforced with iron millscale (IMS) have been carried out. From the results and their analyses, the following conclusions are drawn:

(i) The best and balanced mechanical properties exhibited by the composites occurred at 20 wt. % IMS addition with regard to the specific strength, 58.3 kN.m/kg, 77.8 J impact energy, 160.2 MPa compressive strength coupled with a modest hardness of 146 BHN. These properties were developed at a relatively low density of 2.75 g/cm^3.

(ii) Given its intrinsic tendency to exhibit elastic transformation behaviour during sintering, the addition of IMS particles significantly mitigated the brittle nature of the ceramic matrix composite resulting in enhanced

specific strength and fracture toughness suitable for applications as aircraft fuselage and nose cap material.

(iii) Density is the major factor that determined the composite specific strength and also influences the composite weight regime.

These outcomes have made some contributions in advancing materials processing technology in terms of establishing the powders mixing ratio, adequate compacting pressure of 30 MPa and appropriate sintering temperature of 1350^0C.

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