EVALUATION OF FRACTURE TOUGHNESS OF SINTERED SILICA-NICKEL NANOCOMPOSITES

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Abstract

The micro-Vickers indentation technique is limited to a small area of the specimen surface. It provides a sort of local fracture toughness value, limited to a small portion of the material. But, due to the simplicity of the micro- Vickers indentation test, there has been a steadily increasing interest in its applications. It has been established that classifying on the indentation load, two different types of cracks may form in the Vickers indentation. One type is the Palmqvist crack, while the other is the median type crack which forms in a half penny shape. A Niihara approach is chosen to assess the fracture toughness of silica-nickel Nanocomposites for both type of cracks found in this experiment. The important observation is that with the increase in the amount of nickel, the indentation fracture toughness is found to increased.

Keywords: *Micro-Vickers indentation, fracture toughness, Palmqvist crack, Median crack, Nickel, Niihara, Nanocomposites.*

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1. INTRODUCTION

Silica ceramic has been widely used as a typical engineering material due to its excellent wear resistance, high elastic modulus, and good chemical properties as well as its reasonable cost in the fabrication process.[Tseng et al 2003; Iler et al2004]Recently, Silica has also been used as a biomaterial such as dental materials, total hip prostheses and replacement of various parts of the human body due to its good bio-compatibility as well as its excellent material properties. When brittle ceramic materials such as silica are used in structural application or for machining operations, a high fracture toughness is one of the most important properties to ensure reliability. The main disadvantage of this material was its high-inherent brittleness and hence the performance was not satisfactory. To improve the fracture toughness of Silica ceramic, many researchers have focused on the fabrication of composites by the addition of hard ceramic phases such as Si_3N_4 particles, SiC whisker and fiber reinforcements.[Canut et al 2007, Tu et al 2006] However, the wide application of SiO_2 ceramic has been limited because its fracture toughness was not improved remarkably[Xu et al 2005] Thus, as a different approach, there have been many reports on the ceramic/metal composites in which metal particles were dispersed in the ceramic matrix to introduce the plastic deformation mechanism by the dispersion of ductile phases such as Ni, Co and Fe particles[.Dong et al 2009; Xu et al 2008; Chena et al 2006]. They can remarkably reduce the stress concentration in the notches/micro-cracks due to the plastic deformation. Furthermore, the attractive point for making the Silica based composite is its high melting point(1660^{o}C) compared with that of other metallic systems. Here an approach in which Nickel nano particles are incorporated particles into silica matrix which serve as the binder to improve the fracture toughness.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

Silica-Nickel Nanocomposites powder was prepared by sol-gel technique followed by room temperature drying and subsequent grinding. The cylindrical pellet of powder with nominal dimension of 15mm dia. for the measurement of Micro-hardness and a disc for Elastic Modulus were obtained from the ground powder by pressing uniaxially.The samples are then isothermally heated at the temperature of 1250 ^{o}C in a horizontal tube furnace for 2 h. The heating rate employed to reach the desired temperature is 6 ^{o}C/min.
Samples for the indentation toughness measurements were polished with fine aumina powder on the glass until mirror like surface is achieved. Vickers indentations were made at test loads of 100gf, 200gf, 300gf, 500gf, 1Kgf, 2kgf, 5kgf and 10kgf, using a commercial hardness testing machine. The loading time was fixed at 10s. the indentation sizes and the crack lengths were measured immediately after unloading.

The Vickers micro-hardnes were calculated by the following equation;

$$Hv = 463.6\ P/a^2\ (Gpa) \qquad (1).$$

The Elastic Modulus of the sample was measured using Dynamic Elastic Properties Analyser(DEPA).

The identification of the type crack is essential to the evaluation of the fracture toughness by the indentation micro fracture techniques. A number of similar equations have been proposed to calculate the fracture from the indentation crack length. These have been summarized by ponton et. al[Pontoon et al 1989] and by Li et. al [Li et al 1989] They can be categorized by three different approaches. Palmqvist cracks are addressed as either a semi-elliptical crack[Palmqvist et al 1957; Niihara et al 1983] or a 2-dimentional through crack[Sheety et al 1985].

One approach considers median crack as half penny shapes [Lawn et al 1975; Lawn et al 1975; Lawn et al 1980; Anstis et al 1981; Japaneses et al 1990; Tanaka et al 1987] A third group of equations is based on empirical curve fitting method [Blendell et al 1979; Evans et al 1979 ; Lankford et al 1982]. The equations can be summarized along with their proposers as follows.

(a) Palmqvist Cracks

Niihara et al [Niihara et al 1982]

$K_{IC} (\varphi / H a^{1/2}) (H/E \varphi)^{0.4} = 0.035 (l/a)^{-1/2}$ ----- (2)

And Shetty et.al [Shetty et al1985],

$K_{IC} = (HP/41)^{1/2} / (3\pi(1-\nu) (2^{1/2} \tan\psi)^{1/2}$ ------(3)

(b) Median crack

Lawn and Swain [Lawn et al 1975],

$$K_{IC} = \frac{(1-2\nu)(nH)^{1/2}}{2^{1/2}\pi\xi} (p/c)^{1/2}$$ ----(4)

Lawn and Fuller[Lawn et al 1975],

$K_{IC} = (1/ \pi^{3/2} \tan\psi) p/c^{3/2}$------------(5)

Evans and charles [Evans et al 1976],

$K_{IC} (\varphi / H a ½) (H/E \varphi)^{0.4} = k (c/a)^{-3/2}$---------(6)

Niihara et.al [Niihara et al 1982],

$K_{IC} (\varphi / Ha ½) (H / E \varphi)^{0.4} = 0.129 (c/a)^{-3/2}$----(7)

Lawn, Evans and Marahall [Lawn et al 1980],

$K_{IC} = 0.028 H a ½ (E /H)^{0.5} (c/a)^{-3/2}$---------(8)

Anstis et. al[Anstis et al 1981],

$K_{IC} = 0.016 (E / H)^{0.5} p / c^{3/2}$-------------------(9)

Japanese Industrial Standard(JIS) [Japaneses et al 1990],

$K_{IC} = 0.018 (E /H)^{0.5} p / c^{3/2}$------------------(10)

And Tanaka [Tanaka et al 1987] ,

$K_{IC} = 0.0725 p / c^{3/2}$----------------------(11)

(c) Curve – Fitting Approaches

Blendell [Blendell et al 1979]

$K_{IC} (\varphi / Ha ½) (H / E \varphi)^{0.4} = 0.055 \log (8.4 a/c)$----------------------------(12)

Evans [Evans et al 1979]

$K_{IC} (\varphi / Ha ½) (H / E)^{0.4} = 10^4$------------------(13)

Lankford [Lankford et al 1982]

$K_{IC} (\varphi / Ha ½) (H / E)^{0.4} = 0.142 (c/a)^{-1.56}$-----(14)

In the above equations φ is the ratio of the hardness to the yield stress (H/σ y), which is about 3, ψ is the half angle of the indenter equal to 68o , n and ξ are dimensionless constants which are equal to 2/π and 2, respectively and x= log (c/a).

It is often difficult to determine the type of cracks only from direct observation, because both types of cracks appear quite similar on the specimen surface. However, as first noted by Niihara et.al [Niihara et al 1982; Niihara et al 1983], a significant differences exists in the relationship of the crack length, l, versus the test load p. For low test loads, the relationship is proportional with a steep slope, but when the load load exceeds the certain value, the slope changes abruptly. The initial low load , portion originates from the Palmqvist cracks, whereas the latter is indicative of the median crack. It is therefore possible to distinguish the two crack types by plotting the crack length l, as a function of the test load P.

3. RESULTS AND DISCUSSIONS

The micro-hardness(Hv) and elastic modulus(E) values of the samples) were determined as mentioned above and results of both micro-hardness and elastic modulus were summarized in the tabular form in the table1. From the table 1 and also Fig 1 & Fig.2, it is observed that the values of micro hardness and elastic modulus depicting an increasing trend as nickel content increases.

To evaluate fracture toughness, it is essential to distinguish the palmqvist and the median crack types. The relationship of crack length(l) and Indentation load(p) for the four different Silica-Nickel nanocomposites are summersized in table 2 for synthesized silica -5wt% Nickel, 10wt% Nickel and in table 3 for synthesized silica -15wt% Nickel & 20wt% Nickel and are depicted in Fig 3-6 From the Figs, it is apparent that the relationship possesses a distinct knee of transition at critical test load 10.1 N, 20 N, 48 N and 49.1 N respectively.

Distinct linearity exists in the region where load, P is less that of the knee. In the regions higher than the critical load, linearity of data is not as good. Therefore, below this load, crack region is palmqvist and above is median crack region. Using equation of Nihara et al [eq2] and Nihara et al [eq. 7] fracture toughness for above samples were evaluated both for palmqvist and median type of crack respectively.

The fracture toughness calculated and tabulated in table 4 and table 5 of the samples and are depicted as the function of test load P in Fig 7 & Fig 8 respectively. These results exhibit a range of values from 1.76-3.64 Mpa m $^{-1/2}$ for the samples. The results indicate that the fracture toughness of synthesized silica-nickel nanocomposites increases with nickel content.

With increase in load, the fracture toughness changes hardly (change is in two order of magnitude) whereas the increase in nickel content there is significant change in fracture toughness This is amply clear from the visual inspection of Fig 7 and Fig 8 This is attributed due to the fact that with more of nickel content, the probability of crack being arrested during propagation is more and this makes the fracture toughness of the sample more.

Similar observation was found for median type of crack at high load. Only change of Kic with load is more when compared with palmqvist type of crack.

From Table 6 and the Fig 9 it can also be explained in the same way. Thus, the presence of Nickel in the synthesized Silica-Nickel nanocomposites enhances fracture toughness significantly compared to base synthesized silica (from 30% - 95%). Fig10-13 represent the typical micro crack indentation of samples.

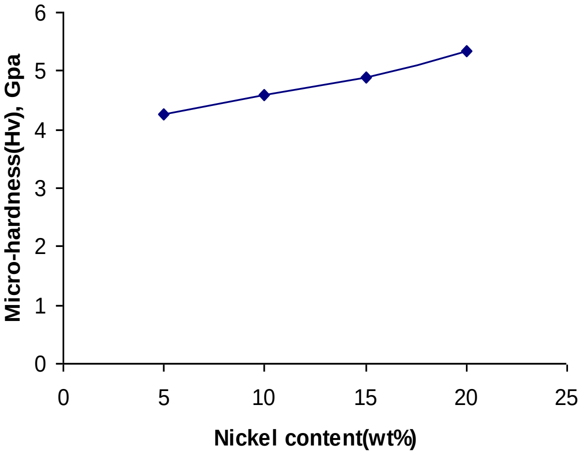

Fig 1 Micro- hardness as function of Nickel content

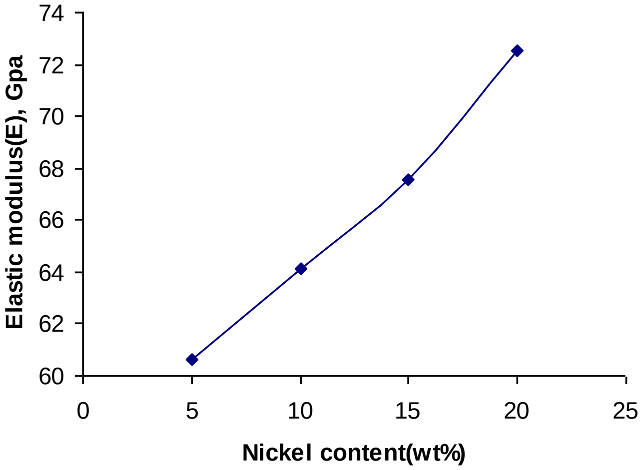

Fig 2 Elastic modulus as function of Nickel content

Table 1 Comparison of micro- hardness and elastic modulus vs Nickel content(wt%)

Samples	**Micro-hardness(.H_v) Gpa**	**Elastic Modulus(E) Gpa**
Silica -5wt% Nickel	4.27	60.60
Silica -10wt% Nickel	4.59	64.10
Silica -15wt% Nickel	4.89	67.55
Silica -20wt% Nickel	5.34	72.55

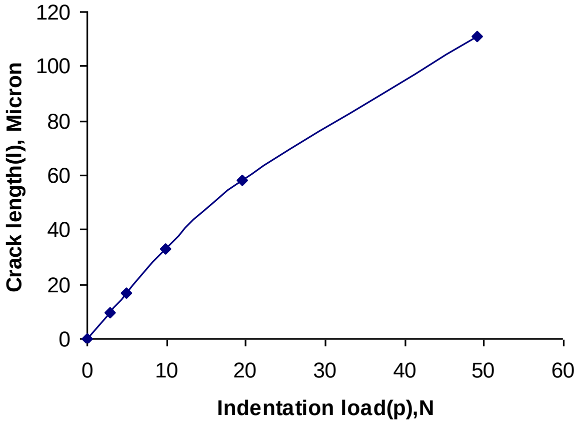

Fig 3 Relationship between Crack length(l) and indentation load(p) for Silica -5wt% Nickel nanocomposite

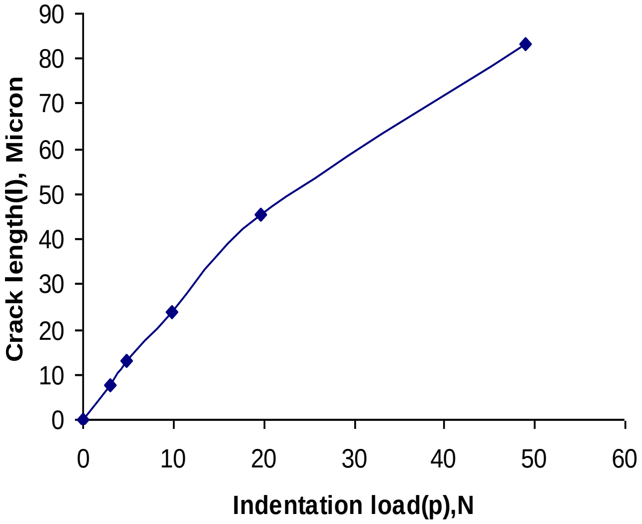

Fig 4 Relationship between Crack length(l) and indentation load(p) for Silica -10wt% Nickel nanocomposite

Table 2 Indentation load(p) vs crack length (l)

Indentation load(p),N	**Crack length(l),µm**	
	Silica -5wt% Nickel	**Silica -10wt% Nickel**
0.00	0.00	0.00
2.94	9.68	7.84
4.90	16.80	12.88
9.81	33.20	23.92
19.62	58.32	45.39
49.05	111.27	83.41

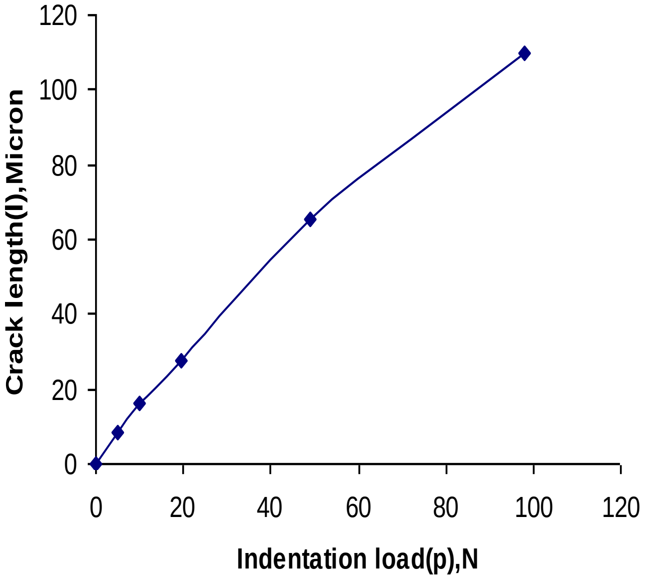

Fig 5: Relationship between Crack length(l) and indentation load(p) for Silica -15wt% Nickel nanocomposite

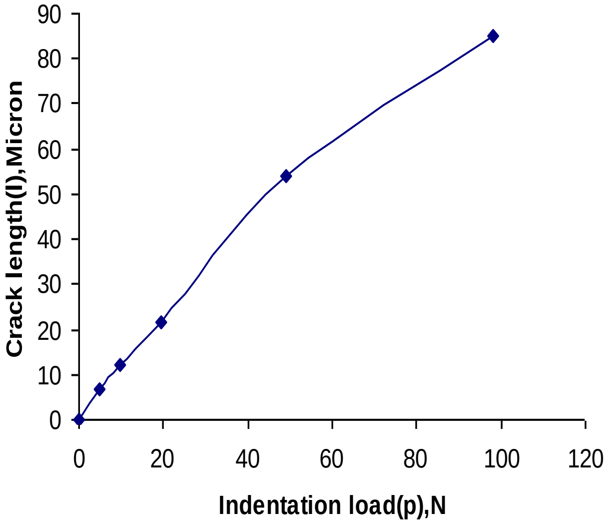

Fig 6: Relationship between Crack length(l) and indentation load(p) for Silica -20wt% Nickel nanocomposite

Table 3 Indentation load(p) vs crack length (l)

Indentation load(p),N	**Crack length(l),µm**	
	Silica -15wt% Nickel	**Silica - 20wt% Nickel**
0.00	0.00	0.00
4.90	8.56	6.80
9.81	16.08	11.95
19.62	27.57	21,48
49.05	65.17	54.21
98.10	109.98	85.07

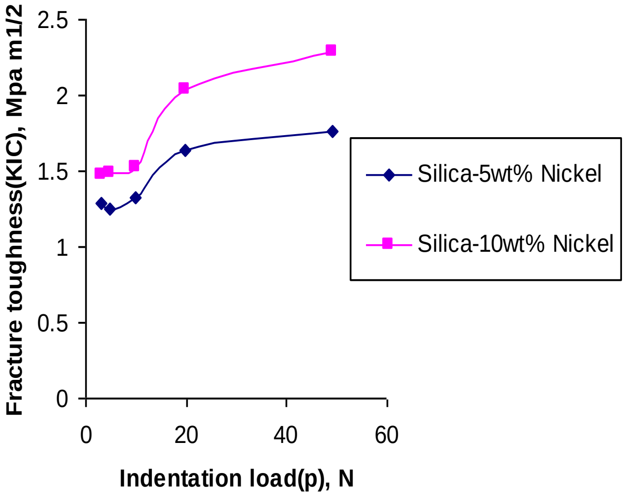

Fig 7 Fracture toughness(Mpa m$^{1/2}$) according to Palmqvist crack model and Median crack model as function of indentation load for Silica -5wt% & 10wt% Nickel nanocomposites.

Table 4: Indentation load(p) vs Fracture toughness(K_{IC})

Indentation load(p),N	**Fracture toughness(Mpa m $^{1/2}$)**	
	Silica -5wt% Nickel Nanocomposite	**Silica -10wt% Nickel Nanocomposite**
2.94	1.29	1.48
4.90	1.25	1.49
9.81	1.32	1.53
19.62	1.64	2.04
49.05	1.76	2.29

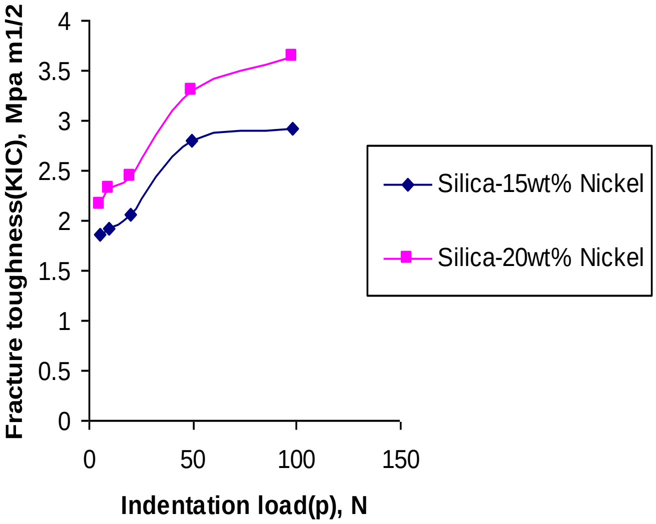

Fig 8 Fracture toughness(Mpa m $^{1/2}$) according to Palmqvist crack model and Median crack model as function of indentation load for Silica -15wt% & 20wt% Nickel nanocomposites.

Table 5: Indentation load(p) vs fracture toughness(K_{IC})

Indentation load(p),N	**Fracture toughness(Mpa m $^{1/2}$)**	
	Silica -15wt%Nickel Nanocomposite	**Silica -20wt%Nickel Nanocomposite**
4.90	1.86	2.17
9.81	1.92	2.32
19.62	2.07	2.44
49.05	2.81	3.30
98.10	2.92	3.64

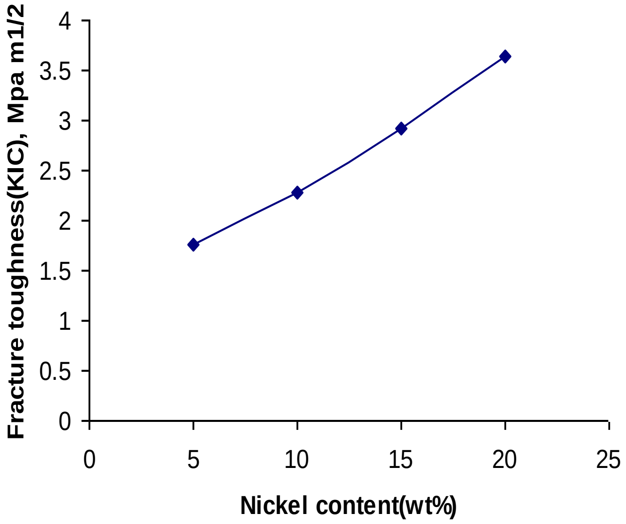

Fig 9 Fracture toughness as function of Nickel content

Table 6 Comparison of Fracture toughness(K_{IC}) vs Nickel content(wt%)

Samples	**Fracture toughness(K_{IC}) Mpa m $^{1/2}$**
Silica -5wt% Nickel Nanocomposite	1.76
Silica -10wt% Nickel Nanocomposite	2.29
Silica -15wt% Nickel Nanocomposite	2.92
Silica -20wt% Nickel Nanocomposite	3.64

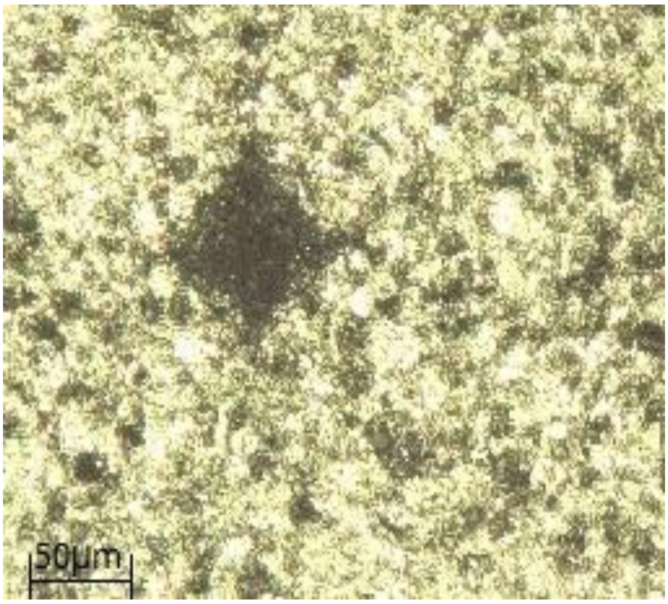

Fig 10 Typical Micro-crack indentation of Silica-5wt% Nickel Nanocomposite

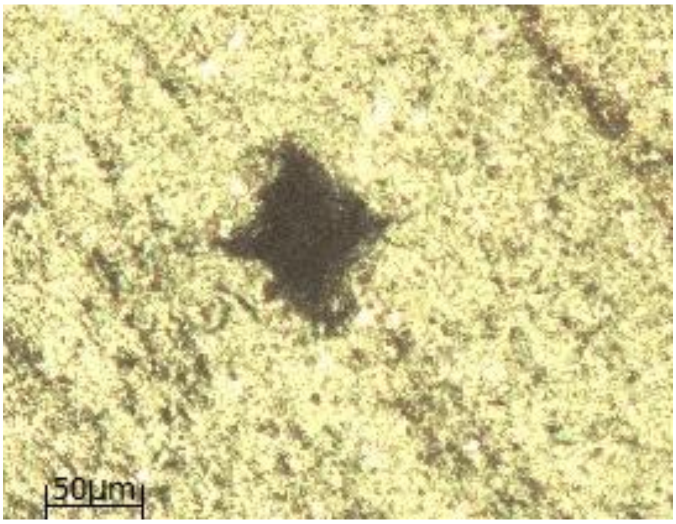

Fig 11 Typical Micro-crack indentation of Silica -10wt% Nickel Nanocomposite

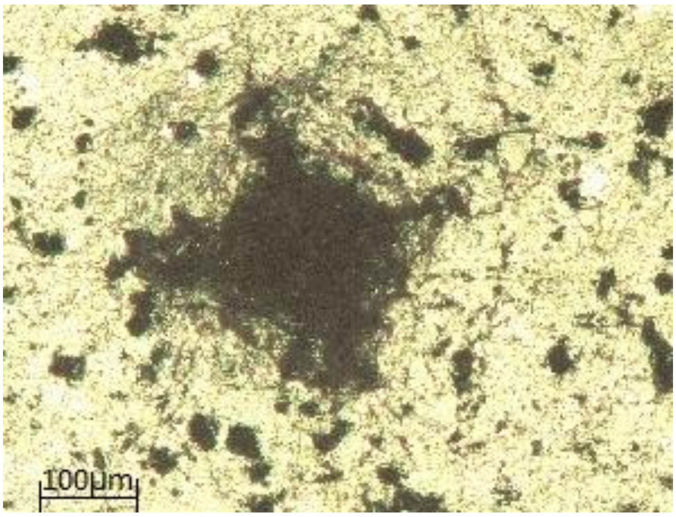

Fig 12 Typical Micro-crack indentation of Silica -15 wt% Nickel Nanocomposite

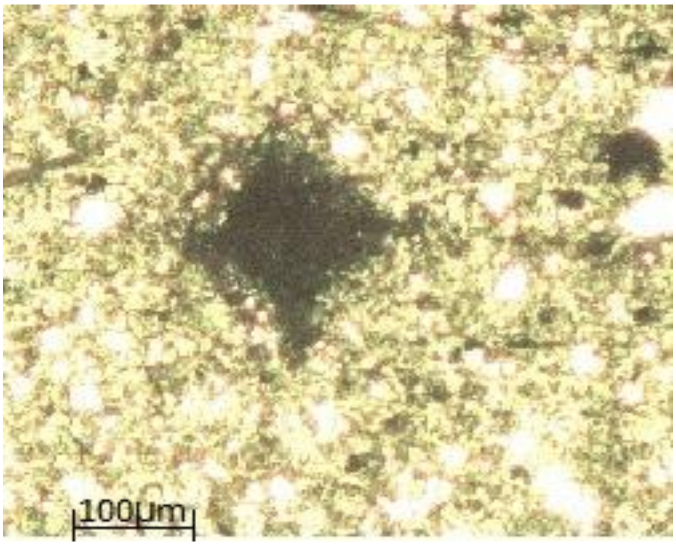

Fig 13 Typical Micro-crack indentation of Silica -20 wt% Nickel Nanocomposite

4. CONCLUSION

1. Micro-hardness calculated from indentations is almost constant regardless of the test load.
2. The Micro hardness of Silica-Nickel Nanocomposites increases as the Nickel content in silica matrix increases and maximum value is achieved in Silica – 20 wt% Nickel Nanocomposite.
3. Fracture toughness of Silica-Nickel Nanocomposites for different compositions enhances significantly as Nickel content in the silica matrix increases and maximum value is achieved in Silica -20 wt% Nickel Nanocomposite.

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