Low velocity drop weight impact behavior of Al_2O_3 - Ni-ZrO_2 and Al_2O_3 - $\text{Ni-Cr}_2\text{O}_3$ nanocomposite systems

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Abstract

1 vol% Ni particulate Al_2O_3 matrix nanocomposites prepared by the heterogeneous precipitation method with ZrO_2 (5 vol%) or Cr_2O_3 (1 vol%) additives were subjected to the low energy drop weight impact tests to compare the behavior of the compositions under low energy impact and to investigate the damage mechanisms. The pure Al_2O_3 , $\text{Al}_2\text{O}_3/\text{Ni}$, $\text{Al}_2\text{O}_3/\text{ZrO}_2$, and $\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$ compositions with the same additive ratios were also produced to make the comparison systematically. Also, the Vickers hardness measurements were carried out and a significant increase in hardness was attained for both $\text{Al}_2\text{O}_3/\text{Ni} + \text{ZrO}_2$ and $\text{Al}_2\text{O}_3/\text{Ni} + \text{Cr}_2\text{O}_3$. The average hardness value around 24.8 ± 1.0 GPa was measured for $\text{Al}_2\text{O}_3/\text{Ni} + \text{ZrO}_2$ and $\text{Al}_2\text{O}_3/\text{Ni} + \text{Cr}_2\text{O}_3$ which means $\sim 15\%$ improvement compared to the pure Al_2O_3 . Between all the compositions, the maximum force (F_{max}) value was obtained for $\text{Al}_2\text{O}_3/\text{Ni} + \text{ZrO}_2$ for 12 J impact energy level (26617 N) according to the low energy drop weight impact test results. Tensile radial crack network formation, cone formation, fracture and crushing of the cone structure were observed as damage mechanisms for all the compositions. The volume of conical frustum structure was evaluated for each composition and the effect of microstructure on possible ballistic performance was also discussed.

1. Introduction

Advanced ceramics have been effectively used in armour technologies for over 40 years because of their lightweight and high mechanical properties [1]. Advances in materials processing and manufacturing technologies make the common use of various ceramics suitable such as silicon carbide (SiC), boron carbide (B_4C), aluminum oxide, Al_2O_3 (alumina) [2]. Among these ceramics, Al_2O_3 is widely preferred due to ease of manufacturing and low cost [3]. Despite the outstanding physical and mechanical properties of ceramics, their brittleness, large scatter of strength are the main disadvantages considering the multi-hit conditions [4]. The mechanical properties of Al_2O_3 can be improved by producing ceramic matrix composites/nanocomposites with different ceramic and metal particle additives such as zirconia (ZrO_2), metal phase (Ni, Cr, etc.) [5, 6, 7]. Also, it is stated in the literature that the formation of substitutional solid solution between Al_2O_3 and chromium oxide (Cr_2O_3) has a remarkable effect on the mechanical properties of the Al_2O_3 - Cr_2O_3 ceramic system [8, 9]. Both manufacturing the composites and the solid solution ceramic systems can be considered to overcome the disadvantages of the material under ballistic conditions.

In view of the armour performance, not only the improvement of the mechanical properties of the material but also the phenomena that occur when a high-velocity projectile strikes an armour target should be considered. The behavior of an armour material under high loading rates ($\geq 10 \text{ s}^{-1}$) is different from low loading rates [10]. When a projectile strikes and penetrates, the rate of deformation encountered by the projectile is much higher than that observed in conventional quasi-static tests [11]. The mechanical properties of materials like compressive failure strength could vary with strain rate [12]. Therefore, it is preferable to measure the basic mechanical properties of armour materials in dynamic loading [13]. In

general, the strength of ceramic materials is relatively unaffected by the strain rate until 10^2 - 10^3 s^{-1} is reached [11]. For ceramics it is argued that crack inertia plays a role, that is, the crack growth rate determines the nature of these materials susceptible to strain rate [14, 15]. On the other hand, there are many problems in testing the behavior of ceramic materials under high strain rate due to their very brittle character and high compressive strength [15].

To develop new armour materials or designing real armour systems, there are some specific tests to understand the dynamic behavior of material e.g. Split-Hopkinson pressure bar test, Taylor test, flyer plate test [11, 16]. Drop weight impact test (DWT) that is given in dynamic low regime range ($5 s^{-1}$ - $10^3 s^{-1}$) is a very common method because of its versatility in imitating the impact behavior of structures. It has the potential to be used in place of time-consuming and expensive low-velocity ballistic tests to investigate the impact damage of materials [17]. It is stated that DWT is a basic method for examining the energy absorption of materials and structures [18]. Also, DWT can be used to determine how much energy is required to completely break a sample, the minimum amount of energy required to start damage to the sample, and how much energy the sample absorbs.

Sherman and Brandon [19] used the DWT for the comprehensive investigation of the ballistic damage mechanisms of Al_2O_3 ceramics supported by steel and aluminum alloy backing. Übeyli et al. [17] examined the low energy impact behavior of Al_2O_3 /aluminum layered composites depending on the areal density and layer combination by DWT. Evci and Gülgeç [20] studied the low energy (3–60 J) impact behavior of the ceramic composite armour system by DWT and the impact behavior with ballistic tests. In their study, 5x10x10 mm Al_2O_3 , SiC, and B_4C were used as a ceramic armour component, and the glass/polyester composite material was used as a composite support material [20].

The failure mechanisms and sequences of damage in ceramic materials can be examined with ballistic tests but it has limitations due to the large number of fractures and fragments that occur in the microseconds time scale during the test. Conversely, DWT allows for particularly low energy impact damage mechanisms to be characterized and it is a relatively simple method [19]. In their previous study, Kafkaslıoğlu Yıldız and Tür investigated the microstructure-mechanical properties relation of the Al_2O_3 -1 vol% Ni nanocomposites prepared by the heterogeneous precipitation method with ZrO_2 or Cr_2O_3 additives to be used in armour applications [21]. In addition to hardness measurements, equibiaxial flexural tests were conducted for strength measurements and the stored energy at failure was estimated by using the theoretical deflection of the cylindrical plate and total crack lengths were measured after failure. The possible fragmentation behavior of each composition was observed, and the ballistic performance of the ceramics were evaluated through the quasi-static tests (elastic modulus, hardness, flexural strength) [21]. In the current study, hardness measurements of the samples produced thicker (~ 4 mm) than the previous study (~ 2 mm) were made and re-examined. The relationship between hardness and density was examined comparatively, considering the new densification conditions obtained. Additionally, low velocity drop weight impact tests of Al_2O_3 -1 vol% Ni nanocomposites prepared with ZrO_2 or Cr_2O_3 additives were carried out for different impact energy levels (5, 8, 10, 12 J). The pure Al_2O_3 ,

$\text{Al}_2\text{O}_3/\text{Ni}$, $\text{Al}_2\text{O}_3/\text{ZrO}_2$, and $\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$ composites and ceramic systems were also tested as well as the binary additive compositions. Drop weight tests were performed not only to compare the behavior of the materials with different compositions under low energy impact but also to investigate the damage mechanisms. Nano Ni and ZrO_2 particulate reinforced ceramic matrix composites and solid solution ($\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$) including ceramic composites were tested to make interference about possible performance against a penetrator under low velocity impact energy through the drop weight test method in this study.

2. Experimental

The pure Al_2O_3 , $\text{Al}_2\text{O}_3/\text{Ni}$, $\text{Al}_2\text{O}_3/\text{ZrO}_2$, $\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$, $\text{Al}_2\text{O}_3/\text{Ni} + \text{ZrO}_2$, and $\text{Al}_2\text{O}_3/\text{Ni} + \text{Cr}_2\text{O}_3$ ceramics and composites were fabricated under the same conditions. While producing the compositions, $\alpha\text{-Al}_2\text{O}_3$ powder (Alfa Aesar, 99.95% purity, 0.25–0.45 μm), Cr_2O_3 powder (Alfa Aesar, 99% metals basis), Y_2O_3 stabilized ZrO_2 (YSZ) powder (3 mol% Y_2O_3 , Inframat Advanced Materials, average particle size < 0.5 μm), nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) (Alfa Aesar, 98% purity), ammonium bicarbonate as precipitant (NH_4HCO_3) (Sigma Aldrich, 99% purity), polyacrylic acid as a dispersant (Darvan 821A, MSE Tech Co. Ltd., Turkey) were used. Also, polypropylene carbonate (PPC) (QPAC 40, Empower Materials, USA) was used as a binder. The heterogeneous precipitation method was used to obtain nano-sized metal particles in the ceramic matrix for the Ni-containing compositions. The heterogeneous precipitation method is an effective method as a coating technique to produce nano sized metal particles in ceramic matrix [6]. Pressureless sintering was used to densitification of the green bodies at 1550°C for 2 h in the air (for the pure Al_2O_3 and $\text{Al}_2\text{O}_3/\text{ZrO}_2$) and in H_2/Ar atmosphere for the other compositions. The detail for the production method was given in the previous study [21]. The amounts of additives were 1 vol% for both Ni and Cr_2O_3 , 5 vol% for ZrO_2 . The volume ratios of additives was determined based on the previous studies and also literature [5, 6, 8]. The sintering temperature was determined according to the preliminary studies that were carried out for the produced compositions by Kafkaslıoğlu Yıldız and Tür, the present literature about the $\text{Al}_2\text{O}_3\text{-Cr}_2\text{O}_3$ ceramic system, $\text{Al}_2\text{O}_3/(\text{ZrO}_2 + \text{Ni})$ and $\text{Al}_2\text{O}_3/\text{Ni}$ nanocomposites [6, 9, 22]. Even though the sintering temperature is above the melting point of Ni and the metal particles tend to grow, a microstructure with intergranular and intragranular Ni particles with sized close to nanometer scale could be obtained [23].

Instead of simply filling the mold as was done in the previous study, a vibrator was used to spread the powders of these samples into the mold before uniaxial pressing. After spreading the powder into the mold with the help of the vibrator, a more homogeneous powder distribution was acquired. This was understood by obtaining a very smooth surface of the powders that appear to be set more tightly in the mold.

Nine disc formed specimens were prepared from each of the compositions with diameters (dimensions vary slightly depending on the composition and the sample) in 28 ± 0.8 mm and thickness in 4 ± 0.2 mm

after the sintering and the grinding process. A single-sided lapping machine was used to grind the specimens equally with an average particle size of 30 μm and 9 μm SiC powder, respectively.

The bulk density of the specimens after sintering was calculated with the direct measurements of mass and dimensions in place of Archimedes' method due to better repeatability and the lower standard deviations. Theoretical densities of the composites were calculated by the rule of mixtures for all compositions to estimate the relative densities from the theoretical densities of Al_2O_3 , ZrO_2 , Cr_2O_3 , and Ni which are 3.98, 6.10, 5.22, and 8.91 g/cm^3 , respectively. Determining the theoretical density for solid solutions is one of the main areas of research in literature [24]. However, for this study, theoretical densities of Al_2O_3 and Cr_2O_3 were used to compare its densification with the pure Al_2O_3 . The hardness of the samples was measured by the Vickers indentation technique with an Instron® Wolpert Testor 2100 equipped with a diamond pyramid indenter under 5 kg load for 10 s and 9 measurements were made for each composition.

Drop weight impact tests were performed on an Instron® Ceast 9340 test machine. Due to the high hardness of the ceramic materials, a 5 mm diameter straight type WC (tungsten carbide) tip was used instead of the common steel tip during the tests. In the tests, the specimens were placed in the machine with vacuum grease by centering on a square alumina (density, 3.80 g/cm^3) support sheet with a width 50 mm and a thickness of 12 mm. This arrangement was preferred to partially simulate the experiment construction with thicker ceramic samples. The alumina support plate was also fitted on a suitably carved tool steel table with 10 mm thickness (Fig. 1.)

Figure 1. The schematic view of the disc-shaped test specimen and the support arrangement.

During the tests, the drop weight was adjusted to a total of 3.404 kg (where the crush head holder mass was 2.5 kg and the crush head nominal mass was 0.904 kg). The sample thickness was entered into the instrument software by adding the thickness of each specimen to the total thickness of the steel plate and the embedded alumina support plate. One specimen of all compositions was impacted at 5 J (drop height of 150 mm, pulse velocity of 1.71 m/s), and if it failed (broken into several pieces), a second specimen is tested at this impact energy level. Then all the specimens were tested at 8 J (240 mm, 2.17 m/s). Tests were repeated at 10 J (300 mm, 2.42 m/s) or 12 J (359 mm, 2.65 m/s) according to whether or not the samples survived. After the tests, the fracture surface analysis was performed by scanning electron microscopy (SEM) (Philips XL 30 SFEG), in secondary electron mode.

3. Results And Discussion

3.1 Densification and hardness measurement results

Al_2O_3 , $\text{Al}_2\text{O}_3/\text{ZrO}_2$, $\text{Al}_2\text{O}_3/\text{Ni}$, $\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$, $\text{Al}_2\text{O}_3/\text{Ni}+\text{ZrO}_2$, and $\text{Al}_2\text{O}_3/\text{Ni}+\text{Cr}_2\text{O}_3$ compositions (abbreviated as A, AZ, AN, AC, ANZ, ANC, respectively) were prepared under the same conditions as the specimens produced for the quasi-static mechanical tests in the previous study as explained [21]. To produce

samples with twice the thickness of the samples produced in the previous study, the vibrator was used for a more compact and homogeneous distribution of the powder in the steel mold before uniaxial pressing. As it is stated in the literature, the steps of uniaxial pressing are as follows; i) shear and rearrangement of the powder particles; ii) fragmentation of the powder particles; and iii) elimination of voids [25]. In ceramic processing, polymer binders are generally added to the system to give some plasticity and to improve the green strength. Also, the vibration allows for better packaging and rearrangement of powders [26]. The use of the vibrator for packaging powders before uniaxial pressing and the following cold isostatic pressing was more effective method than manual shaking for preparing the specimens. So, compared to the previous study, the sintered density of A increased to 97.6 from 95.7, AN to 95.3 from 92.8, AC to 97.8 from 96.7, ANZ to 98.6 from 97.7 and ANC to 96.3 from 94.8 without changing the sintering temperature (1550°C), holding time (2 h) and sintering atmosphere (90% Ar+ 10% H₂). On the other hand, there was no significant difference in the green density values compared to the strength samples in the previous study and again 56-60% values were obtained. In Table 1, the green and the sintered density values of the specimens for each composition are given.

Table 1. The green and the sintered density (%) values of the produced specimens with different compositions and the Vickers hardness values.

Composition	Green density (%)	Sintered density (%)	Vickers Hardness (GPa)
A	60	97.6	21.6 ± 0.9
AZ	60	98.3	21.7 ± 0.9
AN	59	95.3	23.4 ± 0.8
AC	58	97.8	22.6 ± 1.0
ANZ	58	98.6	24.8 ± 1.0
ANC	56	96.3	24.8 ± 1.0

Table 1 also shows the Vickers hardness values of all the compositions. In their previous work, Kafkaslıoğlu and Tür proposed an exponential relation ($H=H_0e^{4.2(1-p)}$) estimate the hardness of Al₂O₃-based composites that increases with the increasing relative density [6]. The relation plot is given in Fig. 2 to compare the hardness values of all the compositions both in previous work and the present work together and to show whether the effect of additives on hardness depends on density [21]. H₀ is the hardness of the fully dense material, p is the porosity. By regression analysis, it was found that H₀ = 23.6 GPa.

The higher hardness value of AZ composites was attributed to the better densification compared to the pure Al₂O₃ in the previous study. In the present study, A and AZ samples have similar hardness values since the difference in density is quite small and their hardness values follows the empirical equation in Fig. 2. A significant increase in hardness was attained for both ANZ and ANC compositions with hardness around 24.8 ± 1.0 GPa which means ~15% improvement compared to the pure Al₂O₃. Even though AN and ANC compositions have lower densities than the pure Al₂O₃, their hardness values are above the solid line and it shows that Ni addition has a positive effect on the hardness of the material.

The hardening effect of Ni particles close to 100 nm size scale is demonstrated and attributed to Hall Petch effect, e.i. small particle sizes limit dislocation motion increasing the yield strength and the hardness of nanometals [27]. It appears that the existence of Ni particles with average particle size near the nanometric value contributes to the hardness for the produced Ni-containing nanocomposites [28]. Ni addition to AZ composites resulted in 14% hardness increase indicating the hardening effect of Ni particles despite the nearly same relative density of AZ and ANZ compositions.

A hardness increase was reported for $\text{Al}_2\text{O}_3\text{-Cr}_2\text{O}_3$ substitutional solid solution in the literature although there are other results that the solid solution effect was not so active for the hardening of the material [8,29,30]. For this study, the hardness of the material increased with the addition of Cr_2O_3 for both the pure Al_2O_3 and AN composite. Even though the relative density of A and AC were similar in the present study, ~5 % hardness increase with Cr_2O_3 addition for the pure Al_2O_3 was obtained. The results indicate the presence of an effect of solid solution formation between $\text{Al}_2\text{O}_3\text{-Cr}_2\text{O}_3$ on the hardness. It was attributed to the differences in ion sizes of Cr^{3+} (0.076 nm) and Al^{3+} (0.068 nm) that generates localized compressive stresses in the crystal lattice in the formation of substitutional solid solution [31]. It is expressed as grain boundary modification and ion-misfit strains promotes the strengthening of the interface [32]. The grain boundary strengthening could also contribute to the hardening of the ceramic by preventing the microcracking and permanent deformation at the grain boundaries.

Fig. 2. The average hardness of the compositions as a function of relative density (filled squares represent the values belonging to the previous work samples, empty squares are belonging to the present study)

3.2 Low velocity drop weight impact test results

Low energy drop weight impact tests were carried out for all the compositions with 9 specimens for each. The experimental results were analyzed in terms of impact forces acting on the specimens, fracture damage mechanisms, and the comparison of fractures based on their composition depending on their impact energy levels. By using the experimental data obtained from the device software, force/energy-time, and force-energy/displacement graphs were drawn. One specimen from each composition were hit at 5 J (1.71 m/s impact velocity, 150 mm drop height), specimens belonging to AZ, AN, and AC compositions failed, and A, ANZ and ANC specimens survived with no apparent damage. Then, a second specimen from AZ, AN, and AC compositions were hit at the same impact energy level and they also failed. So, it was checked whether the fractured state was repeated or not for the same 5 J level. All remaining composition specimens were tested sequentially, adjusting the device for 8 J (2.17 m/s, 240 mm drop height). For an impact energy level of 8 J all of the alumina specimens failed including the specimens that did not break at 5 J before. According to the non-damaged state at an impact energy level of 8 J, the hit was made for an impact energy level of 10 J (2.42 m/s, 300 mm) and 12 J (2.65 m/s, 359 mm), respectively. All specimens of AZ, AC, ANC compositions failed for 8 J impact energy level. For AN composition, only one sample was hit at 8, 10, and 12 J, and this specimen did not fail, and crack formation was not observed. All remaining AN specimens failed at 8 J. Unlike other compositions, 3 of

the specimens belonging to the ANZ composition failed at 8 J, 3 at 10 J, and the remaining 3 at 12 J impact energy level. The summary of whether to fail at different impact energy levels for all composition specimens is given in Table 2. During the experiments, the WC tip was broken, so the tip had to be replaced with a new one, and the relevant sample data was canceled due to the erroneous data received after the tip was broken. In addition, several times have been encountered with the error of not receiving data due to the software error. For these reasons, it was not possible to obtain data from all 9 specimens of the compositions.

Fig. 3 shows the force/energy-time and force/ energy-displacement plots of the pure Al_2O_3 and ANC specimens that failed at 8 J impact energy level. Also, force/energy-time and force/energy-displacement graphs are similar for all the other compositions. When the force-time graphs are examined, an oscillating curve is seen. After the maximum point (F_{max}) was reached, the force decreased following the oscillating curve and eventually become zero. Oscillations in this curve indicate the compressive stresses created in the specimen by the WC tip hit and the tensile stresses reflected from the specimen bottom surface. Compressive and tensile stress waves are thought to interfere. The peaks seen as local minimums correspond to the damping points of the waves. The period of the wave of force-time graphs was calculated as approximately 0.07 ms by averaging the time between peaks of the waves from the graphs. The entire impact event took about 0.8-0.9 ms in all the compositions. When the force/energy-displacement graphs of the specimens are examined, rebound occurs after the maximum deformation point. This means that the elastic energy in the system is recovered. However, it is seen that in all experiments, the targeted impact energy levels (5, 8, 10, 12 J) could not be reached. It seems that the energy difference has gone to the fixing and placement of the underlying system of the specimen (ceramic plate placed on the steel plate) during the experiments. Also, the presence of both the ceramic and steel backing system test setup used for this study makes it difficult to obtain the exact impact energy information of the broken sample simply from the specimen. Some of the energy is transferred to the support system. For this reason, data such as the amount of energy per unit fracture surface and how much energy the material absorbed could not be obtained from the data obtained after the drop weight test for the specimens.

Table 2. Summary of whether to fail at different energy levels for all the composition specimens (S: survived; F: failed specimen).

	5 J				8 J			10 J			12 J		
A		S				F	F	F					
						F	F	F					
						F	F						
AZ		F	F					F					
						F	F	F					
						F	F	F					
AN		F	F					S			S		
						F	F	F					
						F	F	F					
AC		F	F					F					
						F	F	F					
						F	F	F					
ANC		S				F	F	F					
						F	F	F					
						F	F	F					
ANZ		S				F	F	F					
						S	S	S		F	F	F	
						S	S	S		S	S	S	

$F_{\max}$ values obtained for all compositions according to the low energy drop weight impact test results are given in Table 3. As it is seen, the highest average $F_{\max}$ value was obtained for ANZ composition at 12 J energy level (26617 N). In addition, for ANZ composition, the second highest $F_{\max}$ value at 10 J energy level was 25605 N. Generally, all compositions have higher $F_{\max}$ values for 8 J energy level than the pure Al_2O_3 . The high $F_{\max}$ value in the ceramic material is the desired property considering the abrasive and crusher function of this material used in the armour system; the greater the force for certain impact energy, the more penetrator damage if the material is hard enough [20]. The composition of ANZ, with its 24.8 GPa hardness and high $F_{\max}$ values, stands out in all compositions in terms of the possibility of causing more damage to a penetrator.

Fig. 3. The force/energy-time and force/ energy-displacement plots of the pure Al_2O_3 (a and b) and ANC (c and d) specimens failed at 8 J impact energy level.

Table 3. Average $F_{\max}$ values (N) of the drop weight test of all composition at corresponding failure energy levels.

	5 J	8 J	10 J	12 J
A		19566		
AZ	13525	20260		
AN	16906	21620		
AC	15989	21953		
ANC		22014		
ANZ		21468	25605	26617

For the ANZ composition, force/energy-time graphs of the survived specimen for 8 J and the same specimen fractured for 12 J impact energy level are given in Fig. 4 to compare the failed and survived cases. The $F_{\max}$ increased with increasing impact energy level during testing. It is seen that the energy level required for the initiation of damage in the specimen, the propagation of the crack and the material to fracture and lose its integrity are above 8 J for this specimen. The highest $F_{\max}$ value was read for 12 J energy level. In the force-time graph obtained for 12 J, the sudden drops in the peaks are sharper and more distinct. The difference between the failed and the survived specimen graphs may be due to lateral stress wave interferences eliminated in the case of fragmentation of the specimen. At the force-time histories, sudden drops indicated the fracture; if there was no-fail, it did not show sharp drops.

Fig. 4. Force/energy-time graphs of a survived ANZ specimen for 8 J and failed for 12 J impact energy level.

The drop weight impact test is a convenient test in terms of characterizing and understanding the damage mechanism of the material and the order of damage. Sherman and Brandon [19] stated in their study that main damage mechanisms and sequences developed in ceramic material during testing are [17]:

- Tensile radial crack network formation (starts at the lower surface of the material and spreads towards the upper surface)
- Cone formation
- Fracture and crushing of the cone structure

After hitting the penetrator to material, tensile radial cracks occur on the ceramic material. The network of radial cracks that begin at the bottom surface of the ceramic material is the result of tensile stresses that correlate with a regional flexure deformation, leading to a point load applied at the contact interface. A fracture occurs under tensile stress because ceramics have lower tensile strength than compressive strength. Radial cracks (like elliptical cracks) spread over the upper surface. The size of the coarse ceramic fragments is the result of the number of radial cracks and it is the main reason that reduces the material's efficiency at overcoming multiple impacts [19].

Cone crack formation occurs after the formation of tensile radial cracks. The initiation of cone cracking is a slip-dominant mechanism, whereas its propagation is driven by a tensile effect. Cone crack formation occurs after radial crack formation, producing a suitable stress gradient at the edges of the contact zone. The cone cracks propagate as soon as a sufficiently high-stress gradient is produced, and the direction of the cone crack follows the maximum regional tensile stress path. If the compressive stress in the penetrator cannot reach the compressive strength of the material, it will continue to enter the material. Consequently, the material inside the cone will be fractured by pulverizing into various part sizes up to very fine ceramic powder [19].

The formation of radial and cone cracks, fracturing and crumbling of the cone structure determined by visual inspection occurred in all the composition specimens subjected to the drop weight impact test. It is understood that the main stages of the damage mechanism for the respective compositions are not dependent on composition and are valid for all. As an example of these cases, the radial cracks and the cone formation occurring after the drop weight test at 8 J in a specimen belonging to ANC composition are shown in Fig. 5 and Fig. 6. In all the specimens, a complete puncture occurred in the area in contact with the WC tip and the diameter of the hole is approximately the same as the WC tip (5 mm) in the frontal face. Because the impact energy levels are relatively low and close to each other, and the mechanical behaviors differ slightly from sample to sample in ceramic materials, the energy level-broken part number relationship between the compositions could not be established.

Fig. 5. Radial cracks and cone formation by drop weight test at 8 J in a specimen of composition ANC (specimen diameter is 28 mm).

In Fig. 6, a detailed view of the cone formation in a specimen belonging to the ANC composition and a schematic representation of the cone structure, the cone cracks and pulverized parts are given. Considering the ceramic armour applications, the cone-shaped fracture that occurred in the ceramic under impact is believed to be positive. When the penetrator hits the ceramic armour front face, it enables the impact force which is effective in a small area to spread to a wider area in its transmission to the support plate behind the ceramic [20].

Fig. 6. The appearance of cone crack in a specimen of ANC composition after drop weight test at 8 J, schematic, b) remote view, c) close-up view.

The damage mechanisms are common in all the compositions, but the volume of conical frustum may differ depending on the composition. Yamada et al. [33] stated in their study that the volume of the cone is higher, and the energy consumed by surface formation is smaller in transgranular-fractured ceramics than in intergranular-fractured ones. Conversely, there will be usually a combination of the two failure modes, but this will depend on various effects like the packing structure, grain size, the strength of the grain boundary material, loading conditions (static or dynamic) [11,12]. In this study, the volume of conical frustum was estimated from the thicknesses and areas of the holes formed on the frontal and back faces of the specimens for each composition. The graph of the average volume of frustum and its standard deviation by composition is given in Fig. 7. The volume values are estimated by considering the

average conical frustum value for failed samples at all energy levels (8 J for A, 5 J and 8 J for AC, AZ, AN, 8 J for ANC and 8 J, 10 J, 12 J for ANZ) for each composition since there are not enough samples to calculate for each impact energy level. The impact energy applied to the ceramic material may have an influence on the volume of the frustum; however, the difference in measured volumes for different energy levels for each composition is relatively small and justifies the combining of data. As it is seen in Fig. 7, A, AN and AZ have similar average volumes of conical frustum, AC has slightly high average value, but ANC has the highest and ANZ has the lowest value. For the Cr_2O_3 containing compositions, compressive stresses that were generated in the Al_2O_3 lattice due to ion size difference while forming substitutional solid solution may increase the stored energy during failure through residual stresses. This residual stress energy would be released and increase the effective crack driving energy and may result in larger conical frustum volume. ANZ has the highest $F_{\max}$ value and failed at the highest energy level among the compositions. So, it is possible to say that the ANZ consumed more energy while breaking and therefore a smaller volume cone formation was observed in these specimens.

Fig. 8. shows the SEM micrographs of fracture surfaces of all the compositions after the drop weight impact tests. As it is seen, the pure Al_2O_3 (Fig.8a) has a mix (intergranular+transgranular) fracture mode with transgranular mode being more dominant. All the compositions other than ANZ also have a mixed (intergranular+transgranular) fracture mode. In contrast, the crack propagation in ANZ composition was in the intergranular mode. These results coincide with the conclusion of Yamada et al. [33] that the volume of the cone is smaller, and more energy was consumed for intergranular-fractured specimens.

Fig. 7. The average volume of conical frustum structure and its standard deviation depending on the compositions.

During the early stages of penetration into a ceramic armour material, both intergranular and transgranular fracture mode are likely to occur, and intergranular cracking is more beneficial in terms of resistance to penetration because the cracks have to follow along more convoluted pathways around the grains, thus increasing the time for fragmentation to occur [11]. Accordingly, it is possible to predict that ANZ may be a more attractive material against a penetrator damage with intergranular cracking behavior.

Fig. 8. The fracture surfaces of the compositions, a) A, b) AN, c) AC, d) AZ, e) ANC, f) ANZ after the low energy drop weight impact tests (micrograph scale is 5 mm)

4. Conclusions

The low velocity drop weight impact tests were carried out to compare and evaluate the possible behavior under impact conditions of 1 vol% Ni particulate Al_2O_3 matrix nanocomposites produced by the heterogeneous precipitation method with ZrO_2 (5 vol%) or Cr_2O_3 (1 vol%) additive. Also, Vickers hardness of the compositions were investigated by considering densification and effects of additives. Before uniaxial pressing and cold isostatic pressing, a vibrator was used for better packaging and rearrangement of powders for preparing the disc formed specimens. For both ANZ and ANC compositions, ~15%

hardness increase was achieved compared to the pure Al_2O_3 . The hardening effect of Ni particles close to 100 nm size and Al_2O_3 – Cr_2O_3 substitutional solid solution formation that promotes grain boundary strengthening could be responsible for the increase.

After the drop weight impact tests, the maximum force ($F_{\max}$) value was achieved for ANZ composition at 12 J energy level (26617 N). Main damage mechanisms developed in the specimens were common for all the compositions. Tensile radial crack network and cone formation, fracture and crushing of the cone structure were identified. The existence of particles and/or solid solution formation on the ceramic body for such low volume ratios (5vol% for ZrO_2 and 1 vol% for Ni and Cr_2O_3) did not affect the damage mechanisms compared to the pure Al_2O_3 . Although the damage mechanisms are common in all the compositions, the volume of conical frustum differed especially for ANZ. ANZ consumed more energy while breaking and it was resulted in a smaller volume cone formation. All the compositions have a mixed fracture mode other than ANZ with intergranular mode. Intergranular cracking is more beneficial in point of resistance to penetration as the cracks have to follow along more convoluted pathways around the grains, thus increasing the time for fragmentation to occur. Designing composite with intergranular mode can be a good option for armour applications. High hardness and high $F_{\max}$ value are both required when considering the abrasive and crusher role of a ceramic material used in an armour system. So, ANZ composition may come to the forefront with high hardness and high $F_{\max}$ value as a ceramic armour candidate.

Declarations

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Figures

Figure 1

The schematic view of the disc-shaped test specimen and the support arrangement.

Figure 2

The average hardness of the compositions as a function of relative density (filled squares represent the values belonging to the previous work samples, empty squares are belonging to the present study)

Figure 3

The force/energy-time and force/energy-displacement plots of the pure Al₂O₃ (a and b) and ANC (c and d) specimens failed at 8 J impact energy level.

Figure 4

Force/energy-time graphs of a survived ANZ specimen for 8 J and failed for 12 J impact energy level.

Figure 5

Radial cracks and cone formation by drop weight test at 8 J in a specimen of composition ANC (specimen diameter is 28 mm).

Figure 6

The appearance of cone crack in a specimen of ANC composition after drop weight test at 8 J, a) schematic, b) remote view, c) close-up view.

Figure 7

The average volume of conical frustum structure and its standard deviation depending on the compositions.

Figure 8

The fracture surfaces of the compositions, a) A, b) AN, c) AC, d) AZ, e) ANC, f) ANZ after the low energy drop weight impact tests (micrograph scale is 5 μm)