

Experimental analysis, statistical modeling, and parametric optimization of quinary-(CoCrFeMnNi)_{100-x}/TiC_x high-entropy-alloy (HEA) manufactured by laser additive manufacturing

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ABSTRACT

For additional strength increase, 5, 10, and 15% TiC was added to the quinary CoCrFeMnNi high entropy alloy (HEA) at laser powers of 100, 400, and 700 watts while selective laser melting method was engaged in the fabrication. Microstructure, porosity, density, yield and tensile strengths, elongation, and microhardness are among the parameters analyzed. As TiC appreciated from 5 to 15%, the microstructure revealed that the particles were dispersed within the matrix. Also, the addition ensued grain size refinement with increasing particle proportion. Meanwhile, 15% caused an increase in porosity, 0–10% TiC dosage and 100–700 watts laser power led to a decrease in porosity. The same dosage of TiC resulted in a linear improvement in microhardness even as 0–15% TiC ensued gradual reductions in density and elongation. Increases in laser power between 100 and 700 watts were detrimental to elongation but beneficial to density and microhardness enhancement. For composites produced at 100–700 watts laser power, 5–10% TiC increased yield and ultimate tensile strengths whereas 15% TiC decreased strength. For every TiC addition, laser power 100–400 watts generally showed an improvement in strength and microhardness, whereas 700 watts depicted a decrease in strength and microhardness. The optimal input combination was predicted by the developed models to be 15% TiC and 504 watts laser power. Since the deviation between anticipated outcome and validation values for the responses is < 0.05, the models are certified for future prediction of the responses. In conclusion, with 504 watt laser power, the entropy alloy's optimum composition is (CoCrFeMnNi)₈₅/TiC₁₅.

1. Introduction

High entropy alloys (HEAs) are forms of alloy made of the combination of five metals in equal amounts or relatively close amounts. They are craved for in engineering applications because of their excellent mechanical, thermal and electrical properties. They possess strength far higher than the cast and wrought alloys of aluminum, copper, and steel. Hence employed in high-strength application areas of aerospace and

spacecraft [1]. Researchers are working in this area to develop varying types of the alloy. Different processes involved in the design of the HEAs include arc melting, induction levitation melting, high gravity casting, suction casting, solidification casting, mechanical alloying and the recent additive manufacturing technique [2,3]. Examples of developed alloys are AlCoCrCuFeNi, AlCoCrCuNi, CoCrCuFeNi, CrCuFeMoNi, VNbMoTaW, AlCrCuFeNiTi, Al₂₀(TiVCrMnFeCoNiCu), CoCrFeMnNi (cantor alloy) and so on with different phases [2]. There are many

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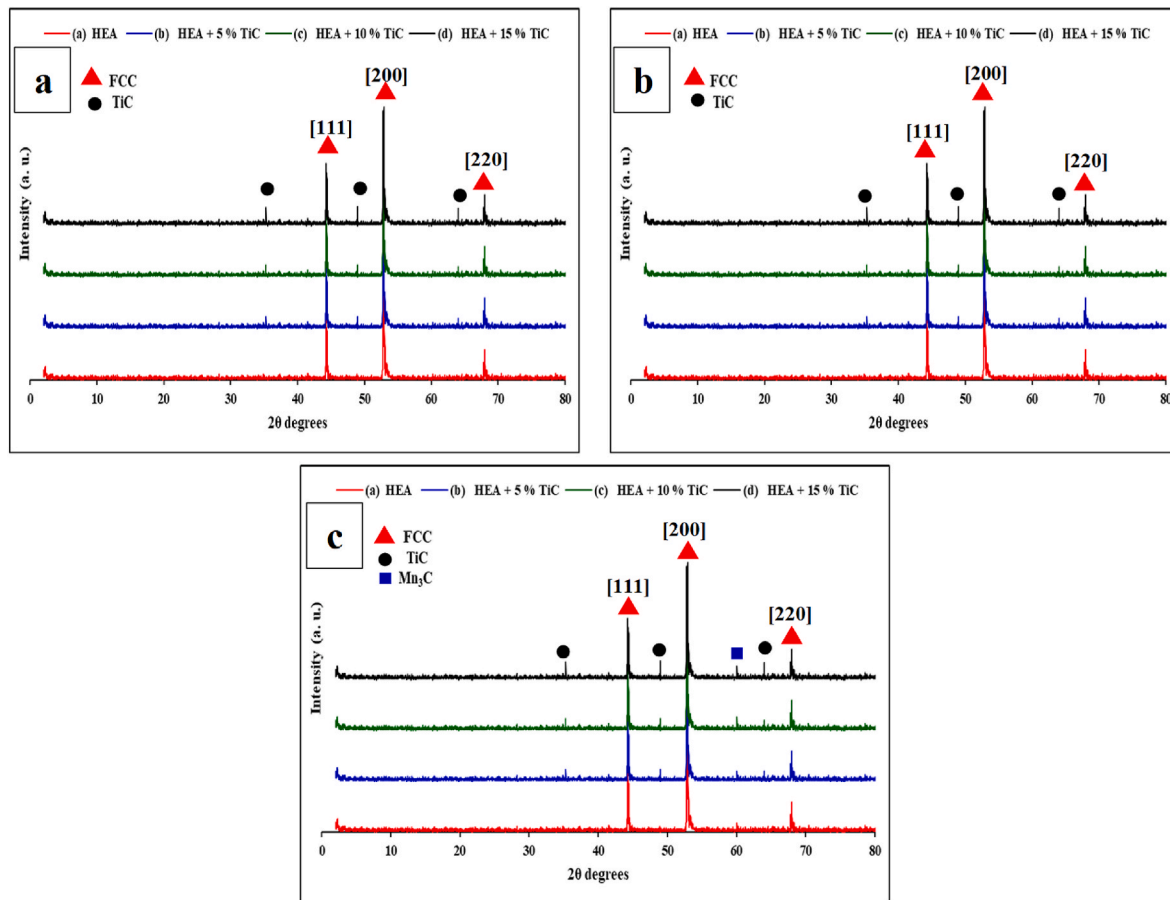


Fig. 1. X-ray diffraction for CoCrFeMnNi-xTiC composites fabricated at laser powers of (a) 100 W (b) 400 W (c) 700 W.

research studies on developing improved HEAs including the addition of ceramic particles in the alloy [4–8] of which one of the most recently used particles is TiC. The particle has played role in the enhancement of HEAs for the alloys prepared by the additive manufacturing process [9]. Zhang et al. [10] introduced TiC in Al_{0.6}CrFe₂Ni₂ alloy at 0, 1.25, 2.50, 3.75, and 5.00 wt %. The result showed that hardness increased with an increase in the TiC dosage yielding a hardness of 264 HV, a 73.7% increase relative to the base Al_{0.6}CrFe₂Ni₂ alloy. Yield strength was optimum at 2.5% TiC addition resulting in a 410.4% improvement over the pure Al_{0.6}CrFe₂Ni₂. Zhu et al. [11] evaluated the hardness and wear of FeCoNiCuAl HEA on the addition of TiC particles at 0, 10, 20, and 30%. The outcome revealed that hardness was boosted as TiC increased from 0 to 30% consequently resulting in a progressive decrease in wear rate. An investigation carried out by Amara et al. [12] revealed a linear increase in the yield strength of CrMnFeCoNi/TiC HEA composite at 2.5 and 5% TiC addition.

Hao et al. [13] reported enhancement of hardness, yield, and ultimate tensile strength of FeCrNiCu alloy reinforced with TiC particles. The optimum performance of the properties was realized at 15 wt % TiC yielding 126.9, 65.9, and 36% for hardness, yield strength, and ultimate tensile strength. Elongation reduced progressively with increasing TiC content engendering a 71.6% reduction in elongation.

Despite the interesting outcomes, there exist gaps in modeling the process for future prediction of responses. This study, therefore, was engaged in the experimental process, development of mathematical models for porosity, density, tensile strengths, elongation, and microhardness, and optimization of the experimental outcomes. Analysis of variance was carried out to determine the significance of the experimental variables which are TiC dosage and laser power. Mathematical models are developed for future prediction of the responses, thereby

reducing the need for large number of experimental runs. Equally, an optimization process was carried out to determine the optimum combination of variables that will yield balanced property responses.

2. Materials and procedures

High purity atomized CoCrFeMnNi powder (20–45 μm) prepared by gas atomization of the ingots obtained through vacuum induction melting were obtained. The TiC powder was of size 10–18 μm (gas atomized). Two parameters varied in this study are TiC dosage and laser power. Variations in TiC are 0, 5, 10, 15 wt % as observed in Wang et al. [14]. 0, 5, 10, and 15% TiC were mixed with the CoCrFeMnNi HEA powder yielding CoCrFeMnNi-0% TiC, CoCrFeMnNi-5% TiC, CoCrFeMnNi-10% TiC, CoCrFeMnNi-15% TiC and melted at laser powers of 100, 400 and 700 W, employing a laser melting 3D printer EOS M 400 of maximum laser power 1000 W, wavelength 1000 nm [15]. The process was done under an argon gas atmosphere (O_2 maintained at < 20 ppm). Mixed powders were passed through the delivery nozzle at 2 rev/min into a molten pool deposited in successive layers on a titanium substrate. The preheated temperature was maintained at 400 $^{\circ}\text{C}$, according to Fiedler et al. [16], layer thickness at 50 μm , hatch space 60 μm , and scanning speed of 800 mm/s were held constant [17]. Through electric discharge machining, samples were cut into dimensions of 120 mm specimen length, gauge 60 mm gauge length, and 10 mm gauge diameter for tensile and 5 $\times$ 5 $\times$ 5 mm for hardness test. The tensile sample was tested employing a Universal testing machine (WAW 1000D) between 20 and 1000 $^{\circ}\text{C}$ and at a loading rate of 10^{-3} s^{-1} . Vickers microhardness was probed on a polished surface with Vickers diamond pyramid (MMT X3) of angle 136° . A load of 5 N was applied for 15 s and the mean result of 5 indentations made was recorded. A scanning

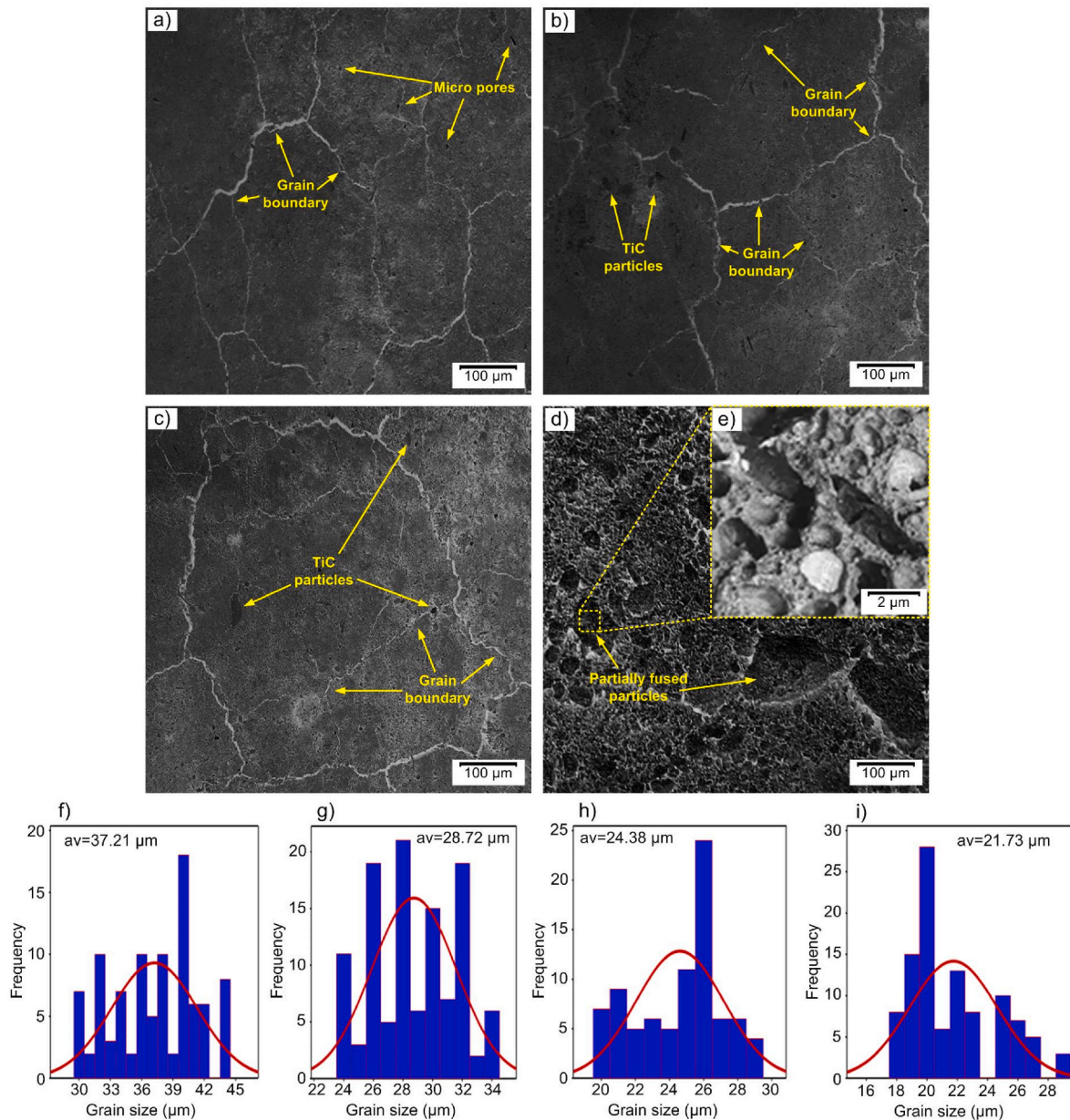


Fig. 2. Morphology of CoCrFeMnNi-xTiC composites fabricated at 100 W with (a) 0% TiC (b) 5% TiC (c) 10% TiC (d) 15% TiC (e) fusion defects observed in 1d; grain size distribution of the composites at (f) 0% TiC (g) 5% TiC (h) 10% TiC (i) 15% TiC.

electron microscope (JSM 7600F Joel) was engaged in the analysis of the microstructure.

3. Results and discussion

3.1. Phase identification

Fig. 1 a, b, and c present the phase diffraction plot (XRD) for the developed CoCrFeMnNi–TiC HEA composites at 100, 400, and 700 W laser power respectively. The reference CoCrFeMnNi HEA identified by the red plot in all the figures depicts majorly a BCC phase typical of CoCrFeMnNi high entropy matrix [18–20]. As observed in the figures, the inclusion of 5, 10, and 15% TiC, showed the presence of TiC reinforcing phases. With increasing TiC content, the peaks of the reinforcing phases increased. No intermetallic phase was observed at 100 and 400 W conditions. As observed in Fig. 1c, there was a presence of Mn₃C phase occurring at 700 W condition. With increasing temperature, there is

higher melting temperature which could be responsible for high-temperature reaction, resulting in the formation of secondary phase.

3.2. Microstructural analysis

Fig. 1 a – e present the morphological images of the HEA specimens prepared with 0–15% TiC at varying laser power conditions of 100 W, while Fig. 1 f – i display the respective grain size distribution.

Fig. 2 a, b, c, d, and e, show the microstructural features associated with 100 W laser power printing of the alloy, while Fig. 1 f – h showcases the grain size distribution at respective TiC addition. The unreinforced specimen (Fig. 1a) presents micropores and grains with the grain boundary highlighted. With increasing 5 and 10% TiC (Fig. 1 b and c), there was a reduction in grain sizes owing to the particle inclusion culminating in grain refinement [21]. Fusion defects (partially or unfused particles) are identified in Fig. 1d, indicating the powders are not

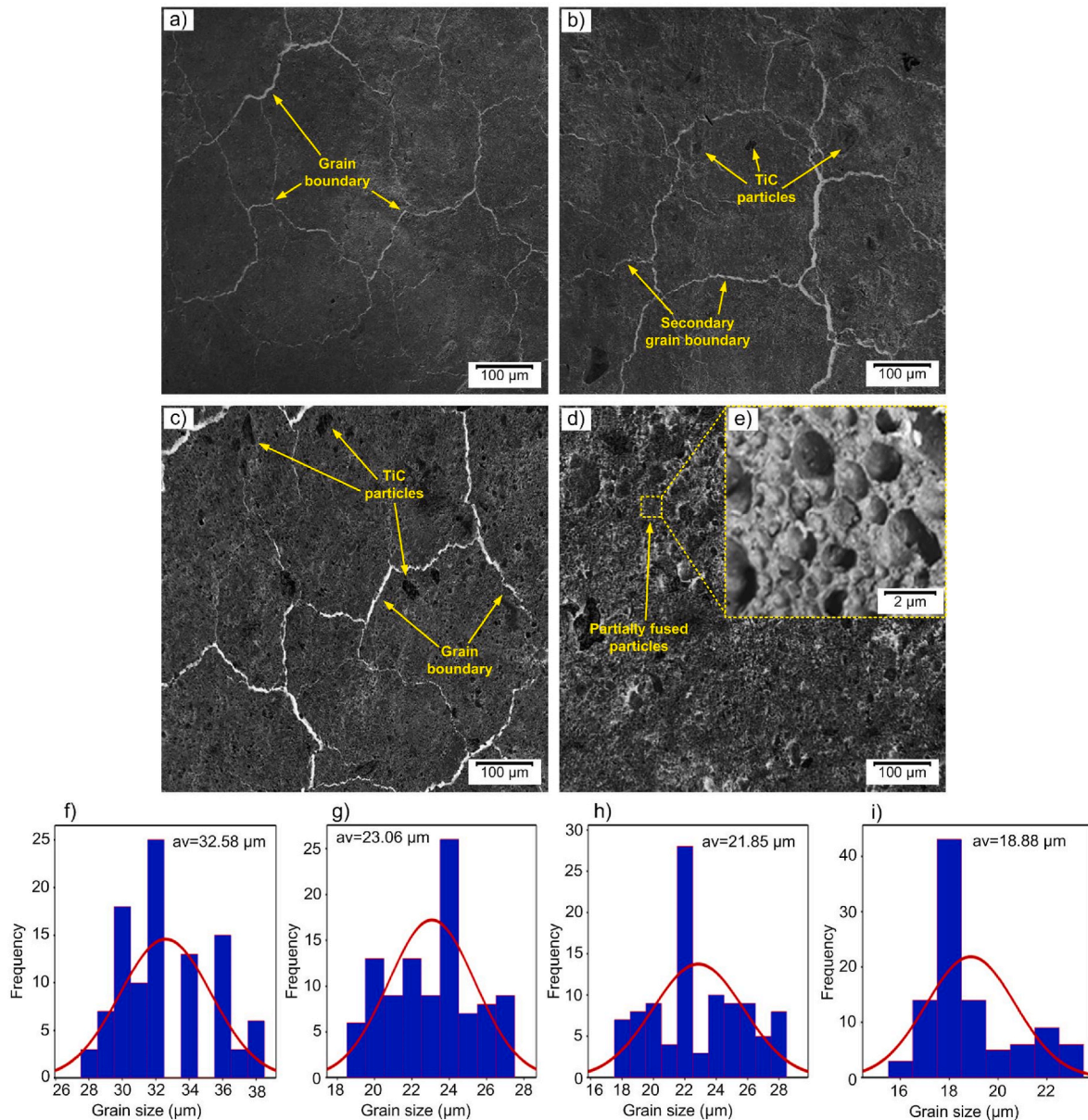


Fig. 3. Morphology of CoCrFeMnNi-xTiC composites fabricated at 400 W with (a) 0% TiC (b) 5% TiC (c) 10% TiC (d) 15% TiC (e) fusion defects observed in 1d; grain size distribution of the composites at (f) 0% TiC (g) 5% TiC (h) 10% TiC (i) 15% TiC.

fully melted/infused, hence engendering a fusion defect either within the layer or at the layers' inter-boundaries. This ensues porosity within the matrix. The observed defect is one of the features recognized during lower melting laser power [22]. Fig. 1e further established the existence of the defect as the microstructure was taken under higher magnification.

Grain size distribution within the matrix at varying TiC content is illustrated in Fig. 2f–i. For the CoCrFeMnNi-0% TiC HEA (Fig. 2f), the grain size ranges from 30 to 44 μm . The modal grain size is 40 μm and the average grain size was evaluated to be 37.21 μm . With the addition of TiC at 5, 10, and 15%, it is observed that there was reduction in the size ranges, modal grain size, and average grain size. The average grain sizes realized at 5, 10, and 15% TiC are 28.72, 24.38, and 21.73 μm respectively (Fig. 2g–i). Therefore, with increasing TiC dosage, there was depreciation in grain size due to inhibition of grain growth. This promotes grain boundary strengthening of the composites [23].

Fig. 3 a - e presents the microstructure of the laser-printed samples at 400 W laser power, even as Fig. 3 f - i illustrates the respective grain size

distribution. The 0% TiC specimen (Fig. 3a) showed majorly primary grains but with reduced size compared with the counterpart at 100 W. Increased laser power showed fast solidification rate giving rise to lower-sized fine grains (Fig. 3b–d). The effect is favorable for the strength of the HEA-TiC composite. In the presence of 5, 10, and 15% TiC, there exist grain size reduction based on the grain refinement with increasing particle dispersion within the matrix. Similar to Figs. 2d and 3d shows fusion defects (partially or unfused particles), depicting the powders are not fully melted at 15% TiC, hence engendering a fusion defect either within the layer or at the layer's inter-boundaries. Observing Fig. 3e taken under higher magnification, there are unmelted and partially melted particles.

Grain size distribution within the matrix at 400 W scanning with varying TiC content is showcased in Fig. 3f–i. For the reference CoCrFeMnNi-0% TiC HEA (Fig. 3f), the grain sizes are in the range of 28–38 μm . The modal grain size is 32 μm and the average grain size was evaluated to be 32.58 μm . This is a 12.44% reduction as compared with the counterpart at 100 W. The assertion can be linked to higher

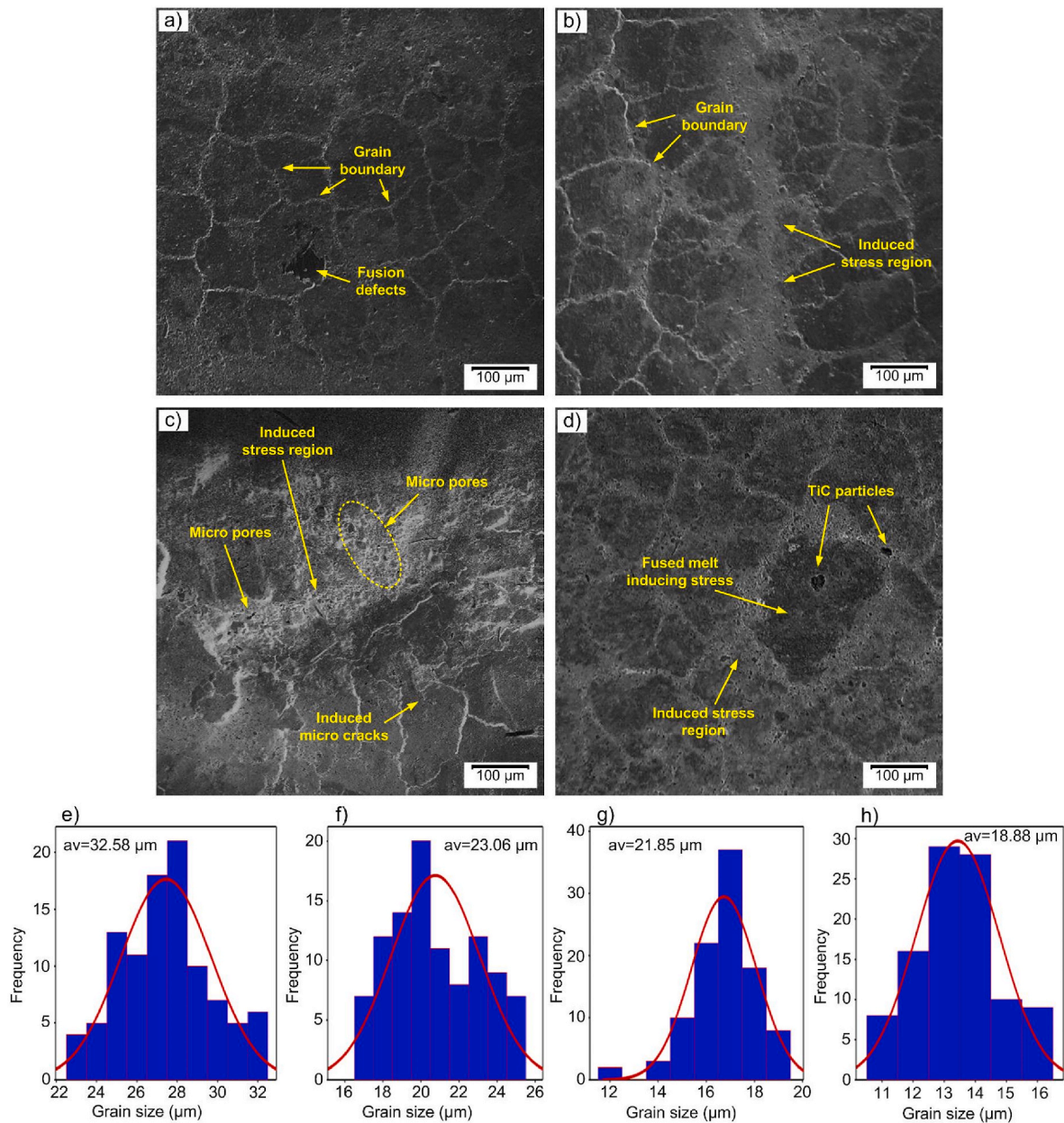


Fig. 4. Morphology of CoCrFeMnNi-xTiC composites fabricated at 700 W with (a) 0% TiC (b) 5% TiC (c) 10% TiC (d) 15% TiC; grain size distribution of the composites at (e) 0% TiC (f) 5% TiC (g) 10% TiC (h) 15% TiC.

temperature gradient which resulted in the faster cooling effect which hindered grain growth. Based on this, it is deduced that variation in laser power contributes to a reduction in grain size. With addition of TiC at 5, 10, and 15% (Fig. 3g, h, i), it is observed that there was reduction in the size ranges, modal grain size, and average grain size. The average grain size realized at 5, 10, and 15% TiC are 23.06, 21.85, and 18.88 μm respectively. Compared with their counterparts at 100 W, the differences in average grain sizes realized are 24.34, 10.38, and 13.12% respectively further affirming the significance of higher laser power in grain refinement.

Fig. 4 a - d represents specimens manufactured at 700 W and melted alongside 0, 5, 10, and 15% TiC as additives. The figures show lower grain-sized morphology. As a result of laser power of 700 W, there is fast cooling. On account of that, the grain sizes are finer, since there is little or no room for grain growth. Fig. 4a-d portrays fine grains smaller than the ones presented under 100 and 400 W. However, there are some defects such as pores (Fig. 4a), induced stress region (Fig. 4b, c, and d),

micropores (Fig. 4c), micro-cracks (Fig. 4c), and incomplete fusion (Fig. 4d). These defects have a negative contribution to the strength of developed composite [24].

Grain size distribution for the composites fabricated at 700 W scanning with varying TiC content is depicted in Fig. 4e-h. For the reference CoCrFeMnNi-0% TiC HEA (Fig. 4e), the grain sizes are in the range of 23–32 μm. The modal grain size is 28 μm and the average grain size was evaluated to be 27.44 μm. In comparison, the average grain size at 700 W is a 26.26 μm, and 15.78% reduction from values realized at 100 and 400 W. TiC contents of 5, 10, and 15% (Fig. 3g, h, i), revealed a further reduction in the grain size ranges, modal grain size, and average grain size. The average grain size observed at 5, 10, and 15% TiC are 20.75, 16.73, and 13.43 μm accordingly. Relative to the counterparts at 100 W, there is a reduction of 27.75, 31.37, and 38.20%. Equally, a refinement of 10.01, 23.43, and 28.87% relative to average grain size was realized at 400 W. It implies that increasing laser power and TiC reinforcement dosage enhanced grain refinement in this study.

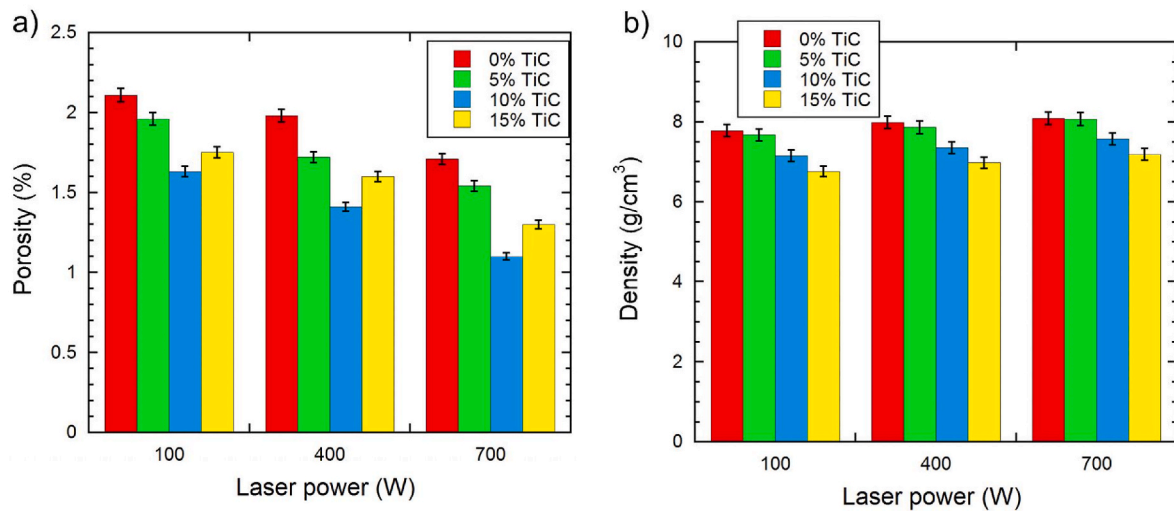


Fig. 5. Relationship between laser power and TiC dosage on (a) porosity (b) density.

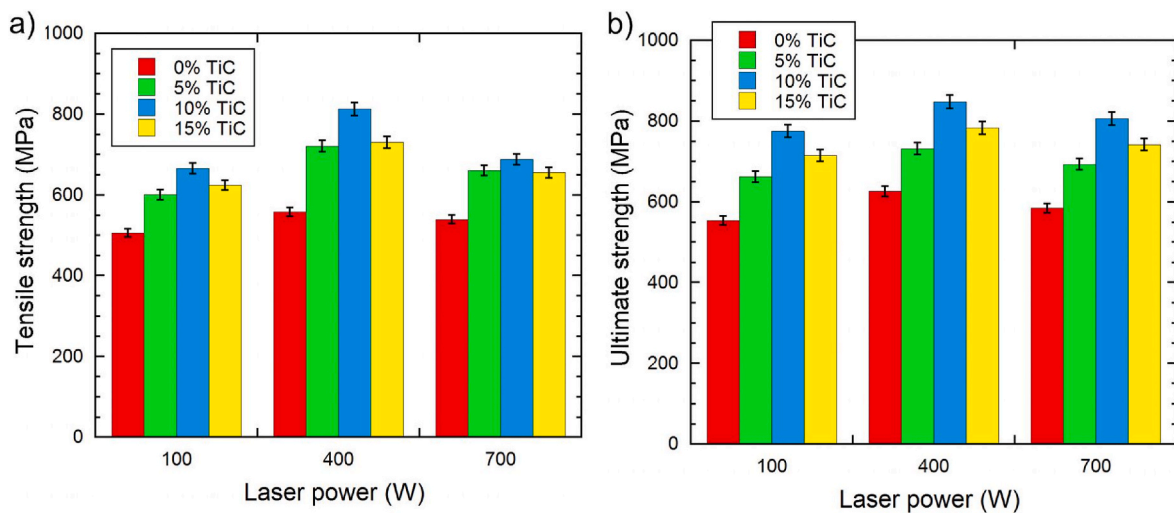


Fig. 6. Relationship between laser power and TiC dosage on (a) tensile strength and (b) ultimate strength.

3.3. Experimental analysis

3.3.1. Porosity and density

Porosity plays important role in the properties of a metal composite. Higher porosity increases interparticle distance and reduces cohesion between particles with a negative consequence on the strength. Lower porosity on the other hand enhances cohesion, promote even stress distribution between matrix and particulate reinforcement, and lower crack propagation tendency within the matrix thereby resulting in strength enhancement. As observed in Fig. 5a, porosity reduced as TiC increased from 0 to 10 wt %. The increase is linked to the infusion of the particles into the melt pool and their ability to fill in inherent pores in the matrix. A similar occurrence is typified in Senel and Gurbuz [25]. In their study, B4C was introduced into aluminum matrix at 1, 3, 6, 9, 12, 15, and 30 wt %. The findings showed that a progressive increase in B4C in the matrix ensued in consecutive decreases in porosity. The same trend was replicated by Meignanmourthy et al. [26]. At 15 wt % of TiC, there was an increase in porosity compared to the value attained at 10 wt % TiC. This is due to increased partial contact existing between adjacent particles. As a result, there is an increase in sites of unfused particles resulting in increased porosity.

The density of the HEA-TiC composite is showcased in Fig. 5b. It is

observed that increasing the dosage of the TiC in the matrix resulted in reduction in density. The reduction is on account of the density difference between the reinforcement and the matrix. TiC powder is lighter than CoCrFeMnNi powder, hence, its increasing proportion showcases a reduction in the density of the composite. Furthermore, with increasing laser power, the densities are noticed to increase on account of increased densification and lower porosity. A similar observation is showcased in Abed and Sabri [27,28].

3.3.2. Yield and ultimate tensile strength

Meanwhile, Fig. 6a reflects the yield tensile strength of the HEA specimens, while, Fig. 6b represents the ultimate tensile strength. The two properties are observed to follow the same trend.

At 100 W laser power, the effect of TiC particles at varying laser power conditions is depicted in Fig. 6a and b. 5 and 10 wt % TiC resulted in 11.9 and 31.6% improvement in yield strength and 20.6, and 42.2% improvement in ultimate strength over the reference sintered at 100 W. A similar study was presented by Li et al. [29] in which 5 and 10 wt % particle reinforcement improved the strength of developed HEA. The feat is linked to the assimilation of the particles in the melt pool yielding better strength performance. According to Hall Petch's theory (expressed in equation (1)), yield stress (T_i) is proportional to $K d^{-0.5}$,

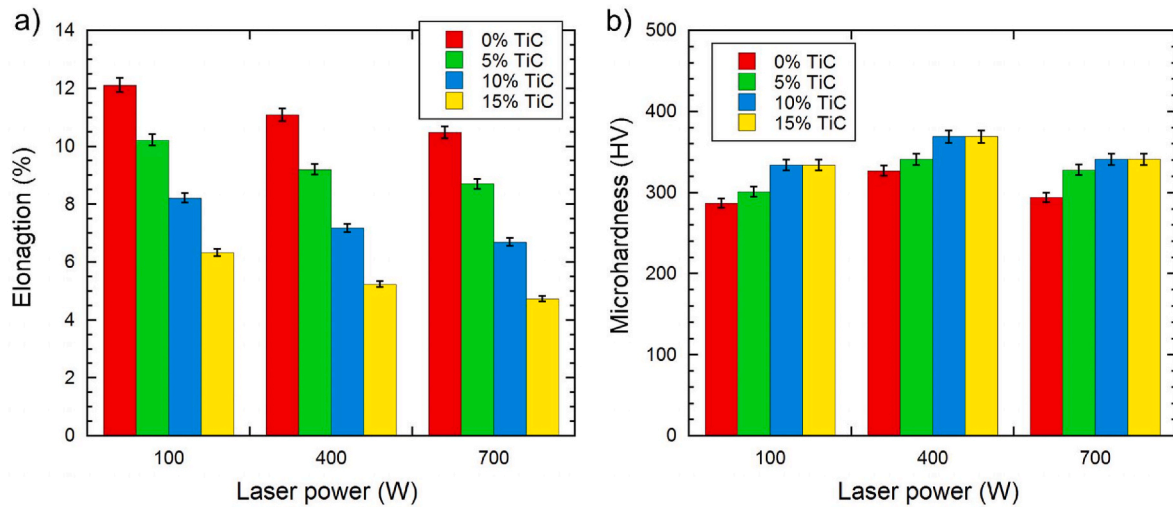


Fig. 7. Relationship between laser power and TiC dosage on (a) elongation (b) microhardness.

where K is the strengthening coefficient of the material and d is grain size.

$$T_0 = T_i + Kd^{-0.5} \quad (1)$$

Deductively, strength value is inversely related to grain size. Lower grain size yields higher strength. With an increase in TiC between 5 and 10%, there is a corresponding decrease in grain size initiating grain boundary strengthening at the microstructural level. Zhang et al. [9] and Zhu et al. [30] explained the role of TiC in refining grain sizes of HEA alloy. CoCrFeMnNi–15%TiC showed lower strength as compared with the CoCrFeMnNi–10%TiC. There is a partial fusion of particles within the microstructure, associated with the lower strength experienced. At 15%, the strength is lower relative to the value obtained when 10% was incorporated, which occurred on account of fusion defects as indicated in Fig. 2d and e. Also, increased porosity is accountable for the strength reduction at 15 wt %.

At 400 W laser power, increasing dosage of 5 and 10% TiC ensued 15.3% and 36.1% increment (yield strength), and 17.8 and 37.2% (ultimate strength) improvement relative to the base CoCrFeMnNi (0% TiC). The observation is attributed to the reinforcing potential of the ceramic particulate. Cui et al. [31] associated the feat with increased bond strength within the matrix. Furthermore, there is the dispersion of the particles within the matrix which aided the matrix in withstanding load. At moderate temperature, TiC was assimilated into the melt pool coupled with a moderate infusion into the matrix. Moreover, it is noted that at 400 W, infusion of 5 and 10% TiC in the HEA matrix yielded higher performance compared with the corresponding values at 100 W. As observed, the grain size at 400 W is smaller than at 100 W causing higher grain refinement, justifying the difference. This is because, at higher laser power, there is a high-temperature gradient and faster cooling preventing grain growth [32]. When 15% TiC was introduced, the outcome revealed a lower value when equated to the value at 10%

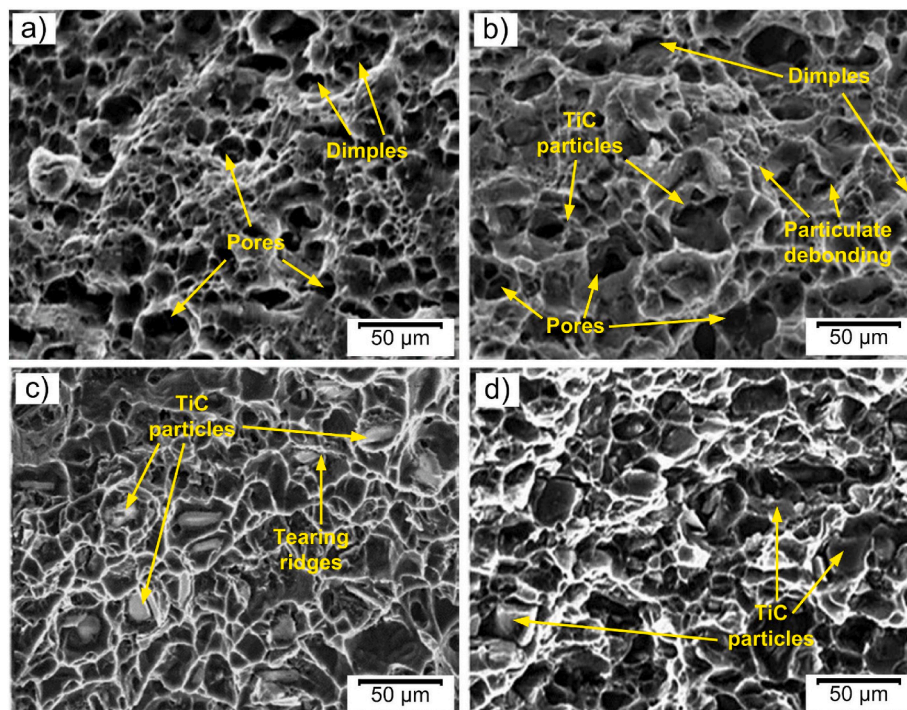


Fig. 8. Tensile SEM fractography of composites printed at 400 W with (a) 0% TiC (b) 5% TiC (c) 10% TiC (d) 15% TiC.

TiC. The fusion defect showcased in Fig. 3d and e is held responsible for the discrepancies.

Upon 700 W laser power application, there exists a 9.5 and 28.4% increment in yield strength when adding 5 and 10% TiC compared with the CoCrFeMnNi (0% TiC). The same dosage of TiC ensued 19.4 and 39.8% improvement in ultimate strength. It is observed in the microstructure that there is existence of cracks and pores. These are defects constituting the strength decrease. Zhang et al. [9] claimed that at high laser power, there is high energy density yielding a high melting temperature. As a result, more powders are melted which may entrap air originating from the original powders. Also, as a result of the high temperature, entrapped gas in the molten pool is unable to escape through the surface, thereby resulting in defects. Owing to elevated temperature on account of the high magnitude of the laser power, the viscosity of the molten pool becomes high leading to reduced cohesion between atoms furthering microstructural defects. Furthermore, as a consequence of the high-temperature gradient, high residual stress is induced within the matrix eventually initiating cracks as observed in Fig. 4c. These are factors held responsible for the lower strength values experienced at 700 W compared with those at 400 W.

3.3.3. Elongation and microhardness

The result indicated in Fig. 7a for elongation revealed that continual rise in TiC dosage in the CoCrFeMnNi HEA ensued linear decrease in elongation for 5–15 wt %. This is true for all composites prepared at 100, 400, and 700 W laser power. The decrease in ductility is on account of an increment in rigidity instilled in the composites by the reinforcement. The trend of elongation reported in this study is in tandem with the findings of Ede et al. [33], Mohamed et al. [34], Sharma et al. [35], Gobalakrishnan et al. [36], Shen et al. [37] in which progressive inclusion of particulate reinforcement of metal matrix engendered reduction in elongation. Increasing laser power from 100 to 700 W showed that elongation reduced with the rise in laser power. This is deduced because the trend of ductility reduced with increasing laser power occurring on account of increased densification. As a result, there is a lower tendency for elongation based on reduction in interparticle distance.

It is revealed from Fig. 7b that at 100 W, 400, and 700 W, there was a progressive increase in microhardness on account of the hard TiC inclusion. A similar report was forwarded by Kong et al. [38] when TiC was infused into Inconel 708 alloy by a similar process. The increase in hardness can be linked to the pinning effect initiated by the particles on account of solution strengthening. It is further revealed that the hardness value at 500 W is higher than at 100 W. This is based on a reduction in grain sizes following Hall-Petch's principle of strengthening. At 700 W laser power, the hardness value reduced compared with the counterpart melted at 400 W based on the effect of the defects observed in the microstructure.

3.4. Fractograph of selected samples

The morphology of the tensile fracture for the HEA-TiC composites is showcased in Fig. 8. Fig. 8a showcased high volume of dimples indicative of a ductile fracture. Pores that are inherent in the matrix are also observed in the figure.

With the increment in TiC dosage from 5–15%, Fig. 8b–d exhibited characteristics of locally ductile and brittle mechanisms. The dispersion of the TiC particles in the HEA matrix is a factor in the ductile and brittle fracture. Decohesion and cracking are the causes of the fracture that was seen in the samples (Fig. 8 b – d). Two factors control the fracture behavior. The first is the disparity in the capacity to withstand strain between the ductile matrix and the hard and inherently brittle reinforcing TiC. The second-factor results from variations in the matrix and TiC particle's coefficient of thermal expansion. The first element encourages the concentration of stress close to the TiC reinforcement particles. As a result, the TiC particles and clusters are more likely to

shatter, which leads to de-cohesion and delamination of the TiC particles from the nearby matrix alloy. In the composite microstructure, simultaneous failure of the reinforcement TiC particles is controlled by the conflicting influences of local plastic limitations, particle size, and degree of dispersion (wider dispersion boosts easy failure). At the contact of the reinforcement and matrix, the second component causes dislocations. By creating effective barriers to the dislocation motion, TiC reinforcement particles minimize the average distance in the composite. The dislocation motion is caused by the interaction of dislocations with TiC particles and other dislocations. Dimple structure is the outcome of the dislocation motion.

At particle/matrix interfaces, void nucleation can be accomplished through particle cracking or interface decohesion. The failure of the ductile metal matrix in between the reinforcement particles is linked to microscopic voids, whereas the reinforcement TiC is coupled with macroscopic voids. The crucial events governing both the fracture and de-cohesion of the reinforcing TiC particles are the causes of macroscopic voids and the resulting shallow dimples on the fracture surface. The presence of hard and brittle ceramic particle reinforcements in the pliable CoCrFeMnNi matrix causes the restrictions. The voids then expand while being affected by the local plastic constraint and the applied stress until a coalescence mechanism is started, which is followed by the complete failure of the specimen. When the void lengthens to the initial intervoid distance, void coalescence takes place. This causes the shattered surfaces to appear dimpled.

Due to the localized localization of hydrostatic stresses brought on by the presence of TiC particles, microscopic cracks start to form and spread during the later stages of deformation in the area of high plasticity. This demonstrates how crucial a role local plastic strains play in controlling damage onset and progression in the CoCrFeMnNi composite microstructure. An example of a mixed-mode ductile-brittle fracture is shown in Fig. 8 b – d. The fracture experienced in Fig. 8 b – d is a mixed mode ductile-brittle fracture.

3.4. Statistical analysis

As performed in Akinwande et al. [39], Akinwande et al. [40], and Adediran et al. [41] statistical analysis was carried out on experimental results to examine the significance of the input variables and their interactions on each property parameter. This was done using the design expert software 13 and effected at a confidence level of 95%. Mathematical models of the second regression order were established for each response for future predictions. Response surface analysis (RSA) entails the design of experiment, analysis of the result, and optimization of process parameters. It appraises the interactions or interplay existing between the independent variables and the effect of such interaction on obtained results. It can be employed in the initial design of experimental as carried out by Ademati et al. [42], Adesina et al. [43], Balogun et al. [44], Adediran et al. [45] in which the design variables are imputed into the design software and an array of input variable combinations representing the experimental runs are realized. As for the other approach, the experiments are initially conducted as deemed fit by the investigators, and the obtained results are imputed into the design software and the result analyzed. The second approach is adopted in this study similar to what was applicable in Akinwande [46], and Akinwande et al. [47]. Also, the analysis employs statistical and mathematical procedures to develop predictive mathematical models from the obtained experimental results. Additionally, analysis of variance is effected on the experimental outcomes to access the significant and percentage contribution of the input variables of the property responses [48]. The variables in this study are TiC dosage and laser power variation. Via the RSA, a mathematical relationship is established between the input factors (TiC dosage and laser power variation) and responses as expressed in equation (1).

$$Y = a_0 + a_1A + a_2B + a_{12}AB + a_{11}A^2 + a_{22}B^2 \quad (2)$$

Table 1

Results of variance analysis on porosity and density.

(a) Porosity						(b) Density					
Source	Sum of Squares	df	Mean Square	F-value	p-value	Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	0.8895	5	0.1779	22.88	0.0008	Model	2.24	5	0.4473	56.31	0.0000
A	0.3557	1	0.3557	45.75	0.0005	A	1.89	1	1.89	237.97	0.0000
B	0.4371	1	0.4371	56.22	0.0003	B	0.2628	1	0.2628	33.08	0.0012
AB	0.0099	1	0.0099	1.28	0.3018	AB	0.0001	1	0.0001	0.0079	0.9322
A ²	0.0645	1	0.0645	8.30	0.0280	A ²	0.0817	1	0.0817	10.28	0.0184
B ²	0.0222	1	0.0222	2.86	0.1420	B ²	0.0015	1	0.0015	0.1894	0.6787
Residual	0.0467	6	0.0078			Residual	0.0477	6	0.0079		
Cor Total	0.9362	11				Cor Total	2.28	11			

A is TiC dosage (%), B is laser power (W).

Table 2

Results of variance analysis on porosity and density.

(a) yield strength						(b) Ultimate tensile strength					
Source	Sum of Squares	df	Mean Square	F-value	p-value	Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	80191.88	5	16038.38	23.45	0.0007	Model	88846.00	5	17769.20	23.38	0.0007
A	32783.91	1	32783.91	47.93	0.0004	A	63180.15	1	63180.15	83.12	0.0001
B	21705.54	1	21705.54	28.96	0.0391	B	2450.00	1	2450.00	3.22	0.1228
AB	52.17	1	52.17	0.0763	0.7917	AB	57.60	1	57.60	0.0758	0.7923
A ²	23972.61	1	23972.61	35.05	0.0010	A ²	16950.08	1	16950.08	22.30	0.0033
B ²	20677.66	1	20677.66	30.23	0.0015	B ²	6208.17	1	6208.17	8.17	0.0289
Residual	4103.86	6	683.98			Residual	4560.92	6	760.15		
Cor Total	84295.74	11				Cor Total	93406.92	11			

A is TiC dosage (%), B is laser power (W).

where A and B represent the input variable in this case TiC dosage, and laser power. Equally, a_1 , a_2 , a_{12} , a_{11} , and a_{22} , are coefficients of terms. In this study, the responses are porosity, density, yield and ultimate tensile strengths, elongation, and microhardness. RSA engages surface plots in the description of the interactive pattern between the experimental variables as obtained from the predictive models. The plot is a 3D view in which the responses are plotted on the Y axis while the experimental variables are plotted on the X and Y axis. Likewise, contour plots are outlines showcasing the predictive value of the responses at varying input combinations. Unlike the response plot, contour plot is a 2D-view connecting varying design points by lines [49]. The two plots were adopted in present study to reveal the dimension of interaction between the experimental variables and the property responses. It is worthy to differentiate between experimental plots (Figs. 5–7) and surface plots. Experimental plots present the trend observed from the observed values while surface plot represent the predicted values obtained from the developed model which were derived from the computation of the experimental data. Furthermore, the experimental plot is a 2D view presenting experimental data in groups, thereby giving a one-directional explanation to the plots and defining the response as a function of only one independent variable/input factor. The surface plot presents all data in a single 3D plot such that the response is expressed as a function of two independent variables. This allows an examination of the

interaction pattern between the input factors and how they affect the trend of the responses.

Additionally, normal plots are employed in the test of the model to indicate whether or not the models showcase high degree of fitness with the experimental data. A perturbation plot is used to examine the impact of the variables at a particular point in the design space. The solution was plotted by altering one element at a time over its range of values while holding the values of the other components constant. The reference point utilized in this investigation is designated 0. Point –1 is the minimum point while +1 is the maximum point.

3.4.1. Variance analysis

Table 1a and b illustrate the result of variance analysis on porosity and density respectively. As observed, the p-values for the input variables (A and B) on porosity and density are <0.05 , hence the contributions are significant. The contributions of interaction A^2 are significant for both responses while that of terms AB, and B^2 are inconsequential on porosity and density.

Tables 2a and b delineate the variance analysis for yield strength and ultimate tensile strength correspondingly. Terms A, B, A^2 , and B^2 are portrayed to reflect consequential influence on yield strength (p-value <0.05), whereas, parameter AB influence is inconsequential (p value >0.05). It implies that TiC loading, laser power, and their square

Table 3

Results of variance analysis on elongation and Vickers microhardness.

(a) Elongation						(b) Vicker microhardness					
Source	Sum of Squares	df	Mean Square	F-value	p-value	Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	54.90	5	10.98	213.09	0.0000	Model	14164.97	5	2832.99	68.01	0.0000
A	50.75	1	50.75	984.93	0.0000	A	10746.82	1	10746.82	257.98	0.0000
B	3.47	1	3.47	67.38	0.0002	B	544.50	1	544.50	13.07	0.0112
AB	0.2387	1	0.2387	4.63	0.0749	AB	28.90	1	28.90	0.6937	0.4368
A ²	0.0507	1	0.0507	0.9840	0.3595	A ²	114.08	1	114.08	2.74	0.1490
B ²	0.3876	1	0.3876	7.52	0.0336	B ²	2730.67	1	2730.67	65.55	0.0002
Residual	0.3091	6	0.0515			Residual	249.95	6	41.66		
Cor Total	55.20	11				Cor Total	14414.92	11			

A is TiC dosage (%), B is laser power (W).

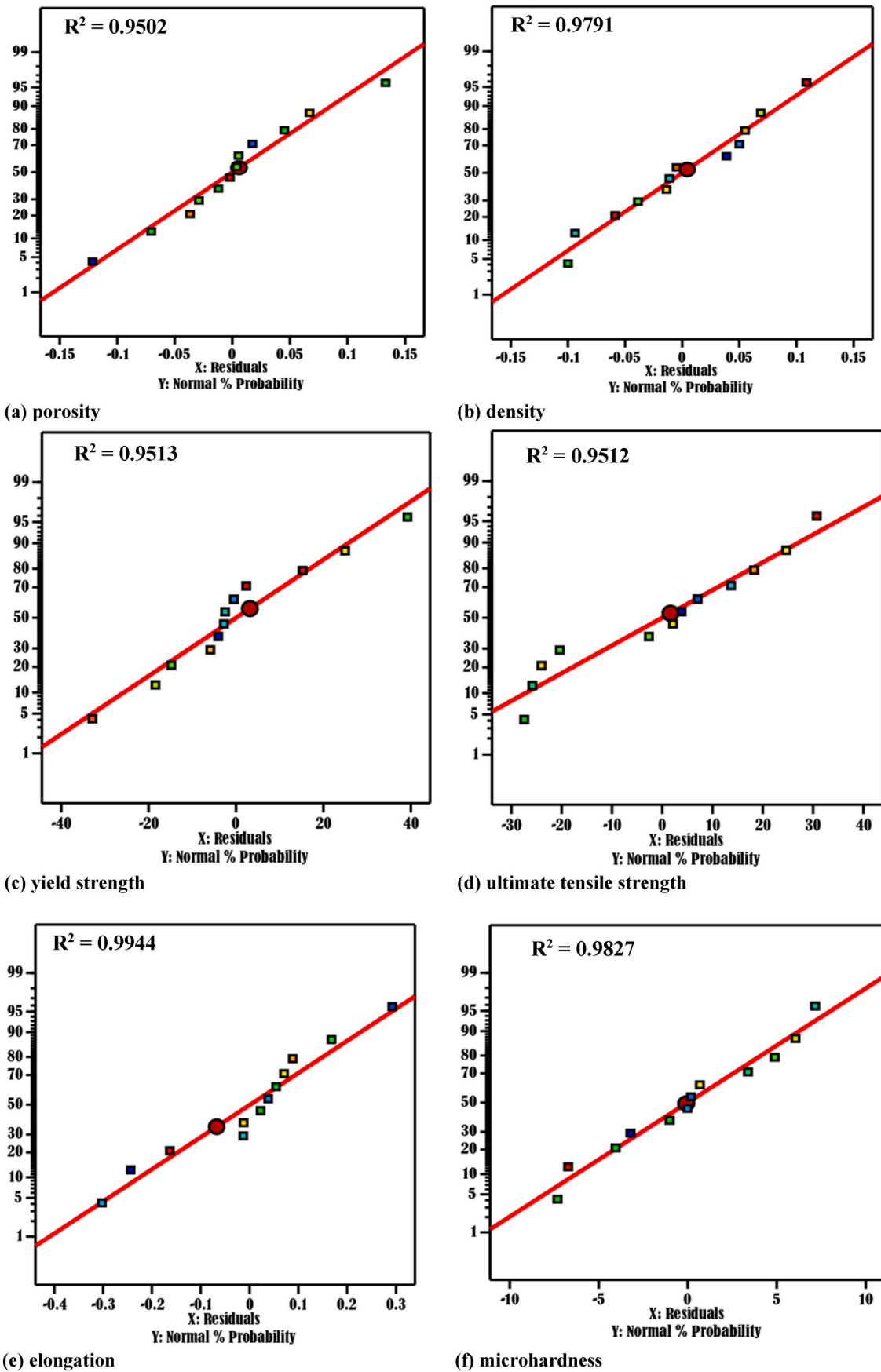
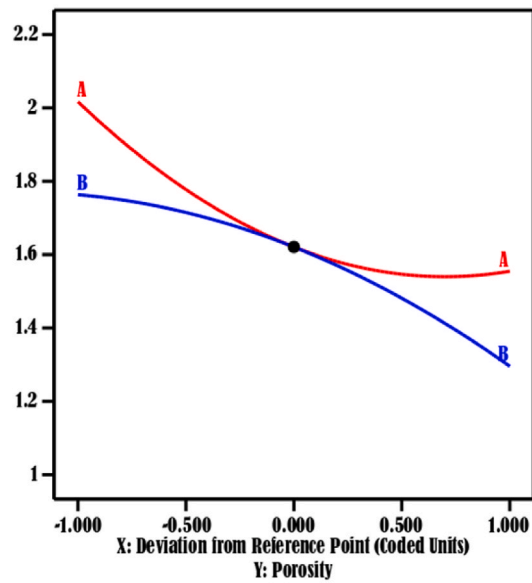
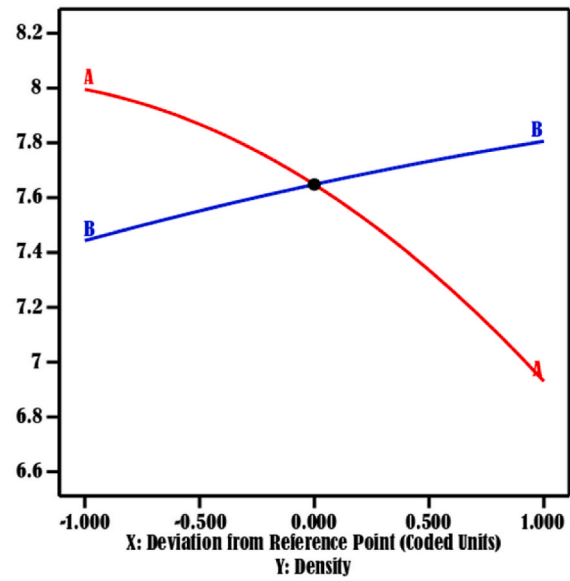


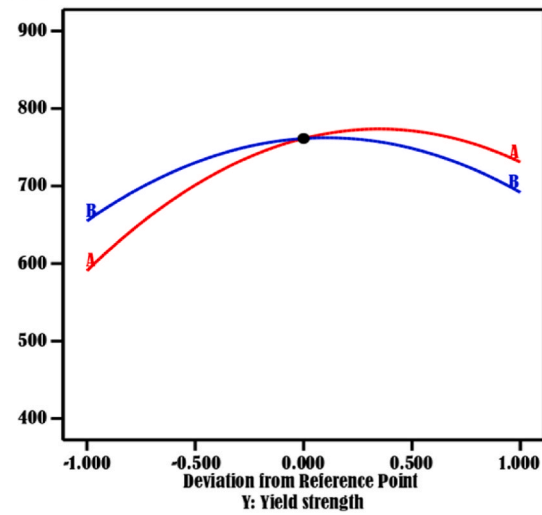
Fig. 9. Normal probability plot for (a) porosity (b) density (c) yield strength (d) ultimate tensile strength (e) elongation (f) microhardness.



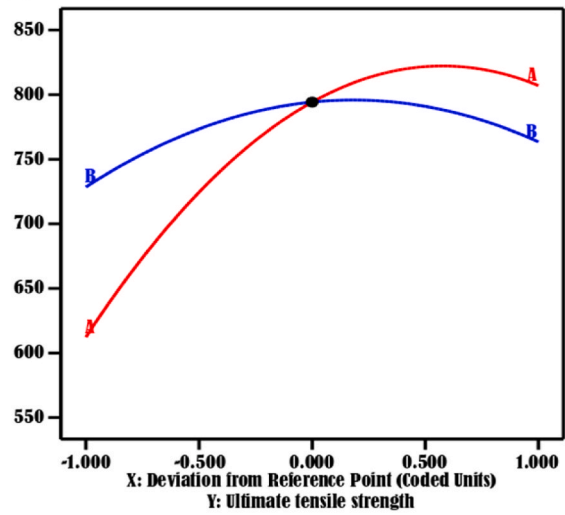
(a) porosity



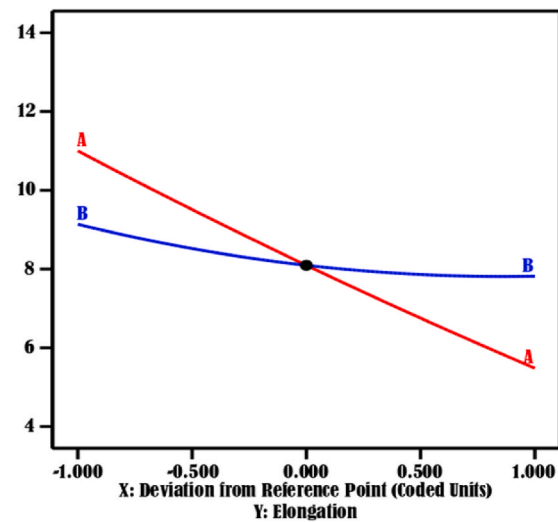
(b) density



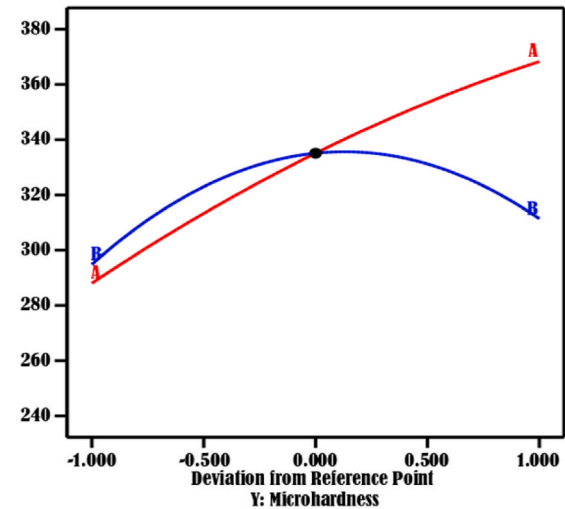
(c) yield strength



(d) ultimate tensile strength



(e) elongation



(f) microhardness

Fig. 10. Perturbation plot for (a) porosity (b) density (c) yield strength (d) ultimate tensile strength (e) elongation (f) microhardness.

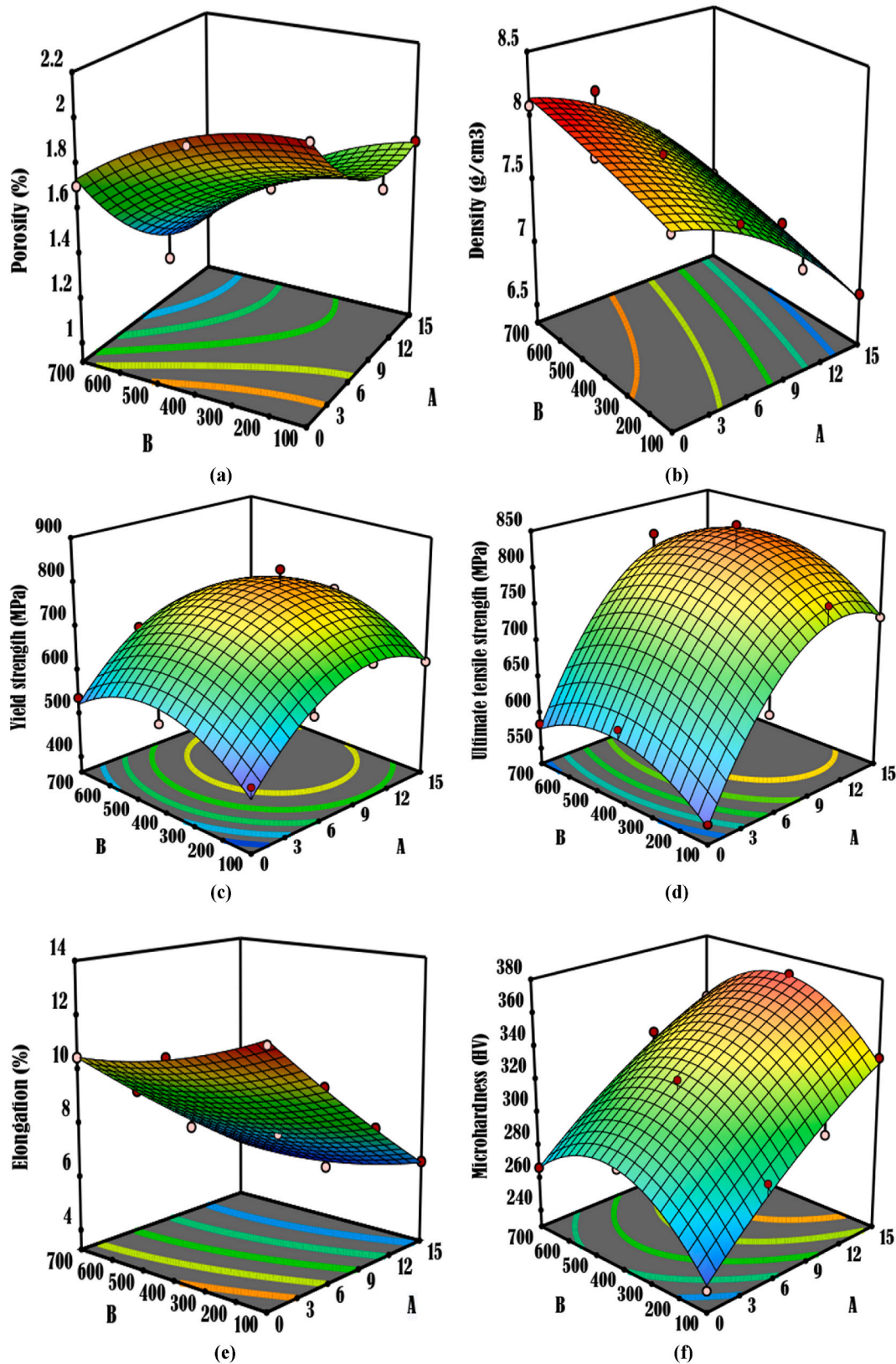


Fig. 11. Surface plots for (a) porosity (b) density (c) yield strength (d) ultimate tensile strength (e) elongation (f) microhardness.

interactions appreciably influence the trend of yield strength. Regarding ultimate tensile strength, parameters A, A^2 , and B^2 presented an appreciable contribution to the response (since p-value < 0.05), conversely, interactions B and AB showed no appreciable contribution

(p-value > 0.05). Therefore, the contributions of TiC dosage, and its square interaction, as well as the square interaction of laser power reflected substantive influence on ultimate tensile strength.

Tables 3a and 3b presents the result of variance analysis on

elongation and microhardness accordingly. The p-values for the input variables (A and B) are <0.05 for both elongation and microhardness. Implication shows that TiC dosage and laser power showed considerable influence on the trend of both properties. Hence the contributions are significant. The same goes for interaction B^2 for both properties (since p-value <0.05). On the contrary, interactions AB, and B^2 had a marginal effect on both responses (p-value >0.05).

3.4.2. Predictive models and diagnostic scheme

The mathematic models representing the responses as functions of the input parameters are dictated in equations (3)–(8).

$$P = 2.1033 - 0.0664 A + 0.000189 B - 0.00002 AB + 0.002933 A^2 - 0.00000 B^2 \quad R^2 = 0.9502 \quad (3)$$

$$D = 7.7161 - 0.02217 A + 0.00080 B + 0.00000 AB - 0.00330 A^2 - 0.00000 B^2 \quad R^2 = 0.9791 \quad (4)$$

$$Y = 405.0765 + 36.7766 A + 0.85546 B - 0.00152 AB - 1.7878 A^2 - 0.00098 B^2 \quad R^2 = 0.9513 \quad (5)$$

$$U = 508.0056 + 34.8900 A + 0.47522 B + 0.00160 AB - 1.50333 A^2 - 0.00054 B^2 \quad R^2 = 0.9512 \quad (6)$$

$$E = 12.8666 - 0.44807 A - 0.006357 B + 0.000103 AB + 0.00260 A^2 + 0.00000 B^2 \quad R^2 = 0.9944 \quad (7)$$

$$M = 216.7278 + 7.65667 A + 0.32044 B - 0.001133 AB - 0.1233 A^2 - 0.000356 B^2 \quad R^2 = 0.9827 \quad (8)$$

P, D, Y, U, E, and M, stands for porosity, density, yield strength, ultimate tensile strength, elongation and microhardness respectively.

The models for the property parameters; porosity, density, yield strength, ultimate tensile strength, elongation, and microhardness are represented by equations (3)–(8) respectively. From Tables 1–3, the p-value for each model are <0.05 , indicating the models are significant. The assertion corroborates the coefficient of correlation R^2 for the respective models which is >0.95 . It is affirmed that the models possess high fitness with the data with marginal variation, hence capable of representing the experimental outcomes. Therefore, there is a good synergy between the predicted values and the experimental values in that the error is marginal.

Fig. 9 a, b, c, d, e, and f display the diagnostic plots for porosity, density, yield strength, ultimate tensile strength, elongation, and microhardness accordingly. The residuals are mapped along the mean line showcasing that the errors have a normal distribution, reflecting a high degree of fitness between the model and the data. Moreso, the coefficient of correlation R^2 for all the responses is >0.95 affirming a good relationship between the models and the observed data.

The perturbation plots for porosity, density, yield strength, ultimate tensile strength, elongation, and microhardness are identified in Fig. 10 a, b, c, d, e, and f respectively. The plots reflect the mean resultant interactive sequence of the variable factors with respect to the responses.

As par porosity and as observed in Fig. 10a, factor A trended down between the minimum point (−1) to the center point (0) before experiencing a turning point between 0 and +1 (high). It shows that for all laser power conditions between 100 and 700 W, TiC dosage between 5 and 10% resulted in a reduction in porosity of which between 10 and 15% there was an increment in porosity depicting a “U” shape trend. Factor B showed a progressive reduction in porosity between points −1 and +1 with a restricted fall between −0.25 and +0.25, thereby picturing a decreasing concave trend. It implies that for all TiC dosages, there is a resultant decrease in porosity between 100 and 700 W laser power application.

In Fig. 10b, term A showed a decreasing concave trend between points −1 and +1 passing through 0. In that case, for all laser power conditions (100–700 W), TiC dosage of 5–15% reflected in resultant

reduction in density. Meanwhile term B (laser power) presented an increasing trend between points −1 and +1 depicting a positive linear trend. By implication, 100–700 W laser power presented a resultant decrease in density for all TiC dosage addition.

For yield strength (Fig. 10c), movement from −1 to +0.5 presents an increasing effect of TiC dosage on yield strength. Beyond such a point, the strength is slightly reduced. TiC dosage, therefore, presents inverted “U” shape trend (turning point at +0.5) with a positive resultant effect on tensile strength. By deduction, 5–12.5% TiC yielded resultant improvement in yield strength, whereas, 12.5–15% resulted in strength decrease. For laser power, movement from −1 to 0 is a positive trend whereas, 0 to +1 is negative. The turning point is at the center portraying an inverted “U” shaped trend. Implicatively, for all dosages of TiC (5–15%), 100–400 W laser power is favorable to resultant improvement in yield strength, of which 400–700 W is not favorable.

As represented in Fig. 10d, factor A showed improvement in ultimate tensile strength between points −1 to +0.75, of which between +0.75 and 1.0 there was a slight decrement in strength, thereby displaying an inverted “U” shaped trend. At all laser power conditions, TiC dosage between 5 and 13.75% engendered a resultant boost in ultimate tensile strength, while 13.75–15% kindled slight depreciation. On the other hand, factor B (laser power) displayed an increment between −1 and 0, but depreciation between 0 and +1, hence, portraying inverted “U” shaped trend. Therefore, laser power between 100 and 400 W showed resultant boost to ultimate strength for all TiC dosage addition. 400–700 W laser power showcased a resultant reduction for all TiC dosage addition.

In Fig. 10e, factor A exhibited a consecutive decrease in elongation between points −1 and +1 passing through 0. This avers that for all laser power conditions, 5–15% TiC decreased elongation resulting in a negatively linear trend. Term B (laser power) showed a slightly continuous decrease in elongation between points −1 and +1 delineating a downward convex trend. In such case, 100–700 W laser power kindled resultant decrease in elongation.

Fig. 10f presents the resultant trend for factors A, and B as regards microhardness. Between −1 and +1, TiC dosage showed progressive increase in microhardness rendering a positively linear trend. Hence, 5–15% TiC dosage addition spawns resultant positive impact on microhardness. As for laser power (factor B), there was an increase in microhardness between −1 and 0, of which between 0 and +1, there was decrement in microhardness. Deductively, 100–400 W laser power is favorable for microhardness, whereas, 400–700 W is detrimental.

3.4.3. Surface plot and contour plot

A is TiC dosage, and B is laser power.

Fig. 11 demonstrates the predictive performance plots of the response properties as obtained from response surface analysis. In Figs. 11a and 0–10.7945% TiC exhibited reduction in porosity, meanwhile, 10.7945–15% TiC gave an increase in porosity. 100–410.186 W showed a slight increase in porosity while 410.186–700 W laser power resulted in reduction in porosity. Porosity depends more on the input factors than the interaction between the patterns. In Figs. 11b and 0–15% TiC showed decrease in density, yet 100–700 W laser power increased density. There is an inverse interaction between the two inputs as regards density. The surface plot for yield strength (Fig. 11c) is such that the inclusion of TiC in the range of 0–10.0686% at a laser power range of 100–404.564 W unveiled a boost in yield strength. Nevertheless, 10.0686–15% TiC inclusion at 404.564–700 W laser power spawned decrease in the strength. In the case of the ultimate tensile strength (Fig. 11d), the interplay between 0 and 10.5543% TiC and 100–429.67 W laser power resulted in strength increment. Howbeit, the interaction between 10.5543 and 15% TiC and 429.67–700 W yield a decrease in strength. An increase in TiC dosage in the range of 0–15% outlined a linear decrease in elongation in Fig. 11e. Likewise, 100–700 W laser power showed a continuous decrease in elongation. With respect to microhardness (Fig. 11f), the inclusion of 0–15% TiC in the HEA-

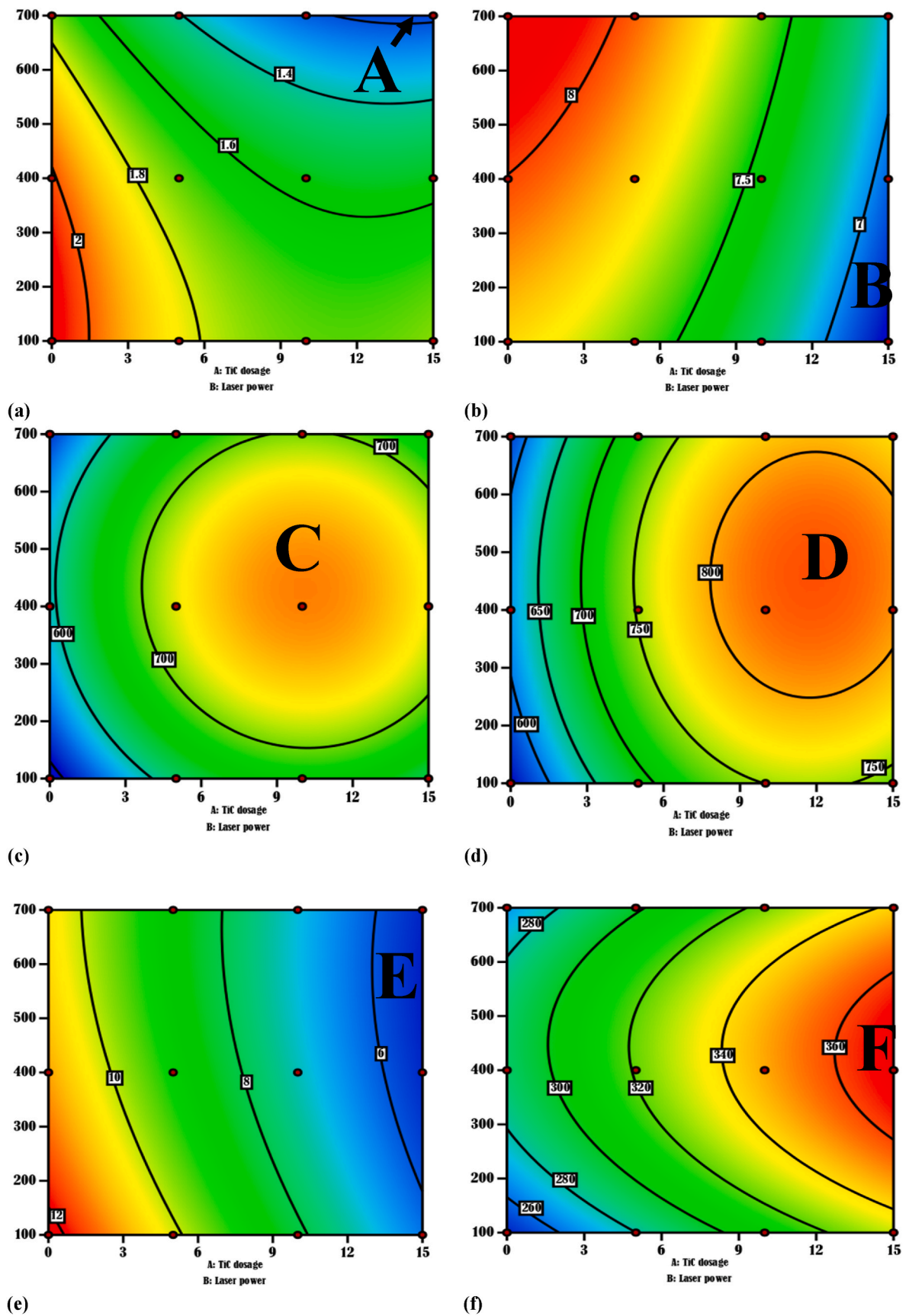


Fig. 12. Contour plots for (a) porosity (b) density (c) yield strength (d) ultimate tensile strength (e) elongation (f) microhardness.

Table 4

Parametric combination for desirable range of outcome.

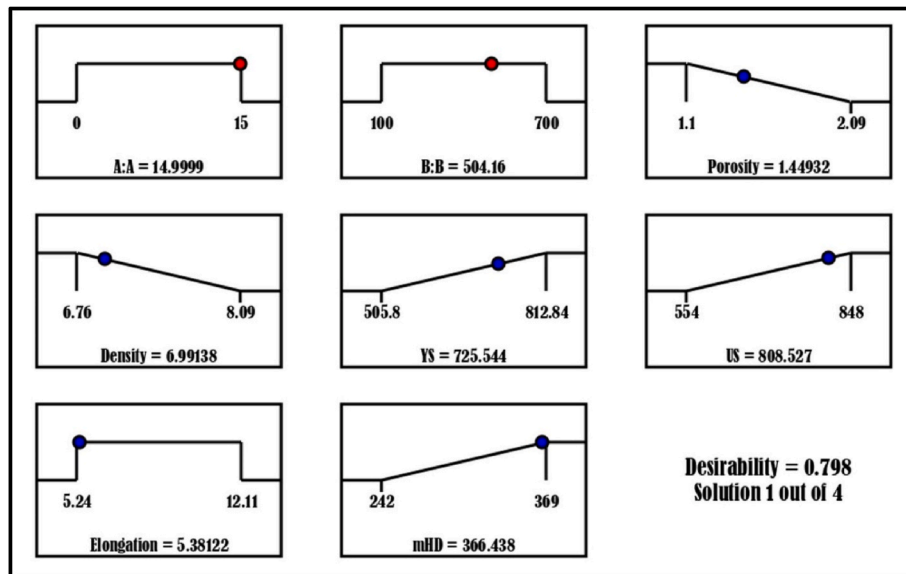
Response	Marked Optimum area	Range values for variables		Predicted range of responses	Predicted desirable value
		A (wt. %)	B (W)		
Porosity	A	11.14–15.0	684.18–700	1.2–1.0%	1.1811%
Density	B	12.46–15.0	100–517.09	7.0–6.5 g/cm ³	6.721 g/cm ³
Yield strength	C	3.67–15.0	152.09–700	700–750 MPa	774.46 MPa
Ultimate strength	D	7.774–15.0	249.03–676.05	800–850 MPa	824.16 MPa
Elongation	E	12.90–15.0	183.06–700	6–4%	5.40%
Microhardness	F	12.59–15.0	271.94–581.79	360–380 HV	368.04 HV

Table 5

Optimization goal and limit ranges.

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
TiC dosage	is in range	0	15	1	1	3
Laser power	is in range	100	700	1	1	3
P	minimize	1.1	2.09	1	1	3
D	minimize	6.76	8.09	1	1	3
Y	maximize	505.8	812.84	1	1	3
U	maximize	554	848	1	1	3
E	is in range	5.24	12.11	1	1	3
M	maximize	242	369	1	1	3

P, D, Y, U, E, M are porosity, density, yield strength, ultimate tensile strength, elongation, and microhardness respectively.

**Fig. 13.** Optimization ramp for the responses.

matrix at a laser power range of 100–410.931 W is favorable to enhancement in hardness. Conversely, 0–15 wt % TiC at 410–700 W laser power ensued a decrease in the microhardness.

The contour plots representing the interplay between TiC dosage and laser power with respect to the responses are showcased in Fig. 12 a–f. Sections marked A, B, C, D, E, and F in the plots are regions in which the combination of the input yields a desirable range of values for the responses. A parametric combination of the factors in yielding desirable range of outcomes is outlined in Table 4. The predicted desirable value for a single response is indicated in the table.

3.4.4. Optimization and model validation

(a) Optimization

The experimental outcomes were subjected to an optimization process through the use of a design expert 13. The goal settings and upper

and lower limits are listed in Table 5.

The optimum condition for balanced performance of the HEA composite realized via response surface analysis is indicated in Fig. 13.

According to Fig. 13, the fitting conditions predicted are 14.999–15% TiC proportion and 504.16–504 W laser power, yielding predicted values of 1.44932%, 6.99138 g/cm³, 725.544 MPa, 808.527 MPa, 5.38122%, and 366.438 HV for porosity, density, yield strength, ultimate tensile strength, elongation, and microhardness accordingly (as obtained from design expert 13 software).

(b) Model validation.

In validating the models, random values were selected for TiC dosage and laser power. The predicted values for the responses were obtained using the model equations, while validation experiments were carried out at the selected conditions, and experimental values were recorded. The values are presented in Table 6. Deviations between the predicted values and validation values were compared and recorded (Table 6). In

Table 6

Table of predicted against validated values at random and optimal conditions (OC).

Parameters	Random conditions					OC
	1	2	3	4	5	
TiC dosage (wt. %)	7.5	12.0	11.5	12.0	13.0	15.0
Laser power (W)	180	220	450	570	600	504
Porosity (%)						
Predicted values	1.7773	1.7176	1.7091	1.7900	1.6932	1.44932
Actual values	1.75	1.70	1.73	1.76	1.72	1.52
% Error	−1.54	−1.02	+1.22	−1.68	+1.56	+4.88
Density (g/cm³)						
Predicted values	7.5082	7.1509	7.3847	7.4309	7.3502	6.99138
Actual values	7.35	7.32	7.53	7.33	7.56	7.04
% Error	−2.11	+2.36	+1.97	−1.36	+2.85	+0.70
Yield strength (MPa)						
Predicted values	700.516	725.709	770.212	747.766	729.654	725.544
Actual values	712.11	714.90	782.61	728.85	718.33	735.2
% Error	+1.66	−1.49	+1.61	−2.53	−1.55	+1.33
Ultimate tensile strength (MPa)						
Predicted values	755.322	792.843	823.204	816.580	810.725	808.527
Actual values	740.08	771.33	850.00	844.90	799.40	827.5
% Error	−2.02	−2.71	+3.26	+3.45	−1.40	+2.35
Elongation (%)						
Predicted values	8.6471	6.7375	5.7300	4.9452	4.4703	5.38122
Actual values	8.82	6.54	5.58	5.11	4.27	4.39
% Error	+2.00	−2.93	−2.61	+3.33	−4.48	−1.84
Microhardness (HV)						
Predicted values	311.831	341.123	354.713	350.085	350.688	366.438
Actual values	323	358	348	361	342	362
% Error	+3.58	+4.95	−1.89	+3.12	−2.48	−1.21

the same manner, a validation experiment was carried out at the predicted optimum condition (OC) and the predicted values and experimented values were compared. The error (%) between the predicted values and the validation values at random conditions 1, 2, 3, 4, 5, and optimum condition OC were noted to be $< \pm 5\%$. Therefore, the optimum parameter for the balanced performance of the composite is 15% TiC and 504 W laser power. Furthermore, the models are statistically adequate in future prediction of the responses.

4. Conclusion

In this study, TiC and laser power were varied in the development of CoCrFeMnNi–TiC composite. The following were arrived at.

- 0–15% TiC and 100–700 W laser power contributed to the microstructural transformation as the variables changed.
- 0, 5, and 10% TiC showed a reduction in porosity, while 15% presented a rise in porosity. Dosage of 0–15% engendered deprecation in density, and elongation, but an increase in microhardness. Meanwhile, 0–10% TiC led to improvement in yield and ultimate strength, whereas, 15% TiC spawned slight decrease in strength. Averagely, there was a boost in strength at 100 and 400 W, of which 700 W depicted a decrease in strength owing to deduced defect in the microstructure.
- Result of variance analysis identified the two inputs had an appreciable effect on the responses. The developed models were fit to represent the residuals. The surface plot showed that the responses were dependent on the relationship between the input variables.
- Optimization process predicted 15% for TiC and 504 W for laser power. The validation experiment carried out established response values of 793.9 and 363.82HV respectively. The deviation between the predicted value and the confirmation value is < 0.05 implying the models are fit for the response forecast. Therefore, 15% TiC and laser power of 504 W for laser power.

Hence, optimal entropy alloy mix is (CoCrFeMnNi)₈₅/TiC₁₅ at 504 W power.

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Sample CRediT author statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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