AlCrFeNiTi HIGH ENTROPY ALLOY PRODUCED BY SOLID STATE PROCESSING

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This paper aims to investigate microstructure and alloying degree of AlCrFeNiTi synthetized by mechanical alloying. Recent studies focus on the HEA utilization in different industries, due to their specific effects that occur, performances and tailored properties.

In order to avoid the contamination that might occur during the process, the powders were maneuvered in a glovebox with controlled atmosphere, but also by using stainless steel balls and vials during the mechanical alloying process.

Samples were taken during the process and analyzed by different methods, thus deciding the best alloying parameters for further use. The results, present a high alloying degree and good homogenization in the final powder of AlCrFeNiTi high entropy alloy.

Keywords: mechanical alloying, high entropy alloys, geothermal, corrosion.

1. Introduction

Compared with traditional alloys [1,2], high entropy alloys (HEAs), which is a new alloy system, tend to form simple solid solution such as FCC and BCC rather than intermetallic compound phases [3,4]. They have high strength, substantial structural stability, good corrosion and wear resistance [5,6].

The good properties of high entropy alloys over conventional materials and alloys promote them in a wide area for the modern industry as metallurgy, automotive, electronics, aerospace. These properties provide the possibility of using HEAs for parts utilized in very aggressive environment and difficult work conditions.

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In this work, the main objective was to synthesize AlCrFeNiTi high entropy alloy by mechanical alloying followed by compaction and sintering. The alloying behavior and microstructure of the obtained HEA was investigated.

A liquid state processing approach tends to be difficult, if elements with highly different melting points are part of the alloy. Mechanical alloying provides the possibility to combine elements on a microscopic scale via cold welding with more homogenous microstructures and better densification characteristics [7, 8].

The HEA was produced by mixing and mechanical alloying high-purity powders and then densified them by pressing and sintering. From thermodynamic calculation of stable phases it was predicted that the microstructure of AlCrFeNiTi HEA consists of BCC [9]. There is a concordance between theoretical structure predictions and the classic criteria of valence electron concentration (VEC). Microstructure analysis of the obtained alloy via SEM and EDAX confirmed the predicted phase.

M. Reiberg et al. has obtained AlCrFeNiTi HEA by mechanical alloying route. They reported a BCC phase with a high Vickers hardness of 839 HV10 and a density of 6.33±0.32 g/cm^3 [9].

2. Materials and methods

The aim of this research was to develop novel and cost effective materials, with superior corrosion-erosion resistance, to counter aggressive environments;

The goal was to produce AlCrFeNiTi high entropy alloy by mechanical alloying that was further pressed and sintered in order to obtain a bulk material.

Due to the very high purity of the elemental materials, the powder manipulation was performed in a glovebox with a low oxygen level (under 3%) and Argon atmosphere.

The solid-state processing was performed by using a Planetary Ball Mill (Pulverisette 6, Frich®), with stainless steel balls and vial. During the milling process, Argon was present, in order to avoid oxidation.

During the mechanical alloying process, samples were collected, and SEM and EDS analyzed by using X-Ray Energy Dispersive Spectroscopy (XEDS) and Field Emission Scanning Electron Microscope (FE-SEM) equipment, in order to observe the evolution of the alloying degree and the mixture homogenization.

The raw powder materials and the final alloy were metallurgical characterized and compared, highlighting the effect of the mechanical alloying effect for the mixture. The metallurgical characterization was performed by using a standard Carney funnel.

The final alloy was pressed by using Walter+Bai AG LFV-300 kN equipment. Several tests were performed, in order to obtain compact samples that can be maneuvered.

The samples were then sintered in laboratory furnace Model Multilab LAC/09, with controlled Argon atmosphere.

3. Results and discussions

For this paper, AlCrFeNiTi was selected, where corrosive and wear resistance properties are to be expected. The material benefits from the cocktail effect and for our experiments we use high purity raw materials of Al, Cr, Fe, Ni and Ti [10, 11].

In Fig. 1 a sample of the homogenized mixture was SEM and EDS analyzed. The results reveal that the particles do not have a uniform shape and size and the composition is confirmed

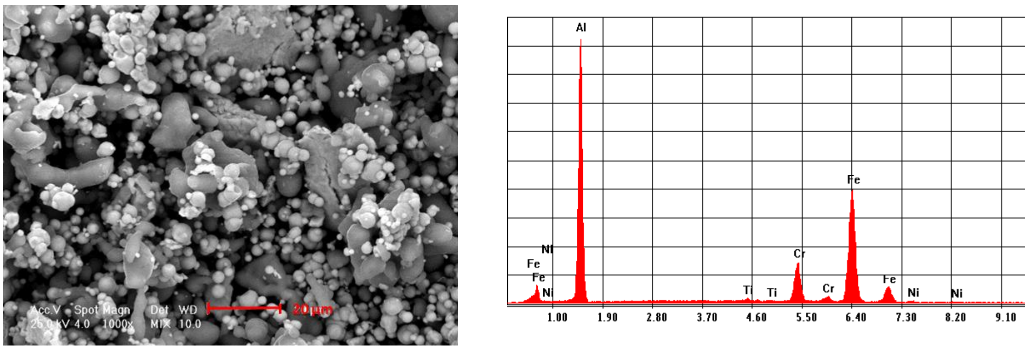

Fig.1 SEM and EDS analyses results for the homogenized sample of AlCrFeNiTi high entropy alloy

For the milling process stainless steel vials and balls were used, with a ball to powder ratio of 10:1 and N-Heptane as a process control agent with a milling time of 30 hours. Wet milling with N-Heptane was preferred due to the good results obtained in previous experiments. Wet milling is preferred for this case, this way preventing the powder to weld on the vial walls and help the powder particles to weld and be crushed again repetitively to ensure a homogeneous mechanical alloying process. The rotational speed used for the mechanical alloying process was 300 rotations per minute.

In Fig. 2 can be observed the microstructure evolution of the AlCrFeNiTi for 5h, 10h, 15h and 20h and can be noticed that the particles were welded and broke and re-welded during the process.

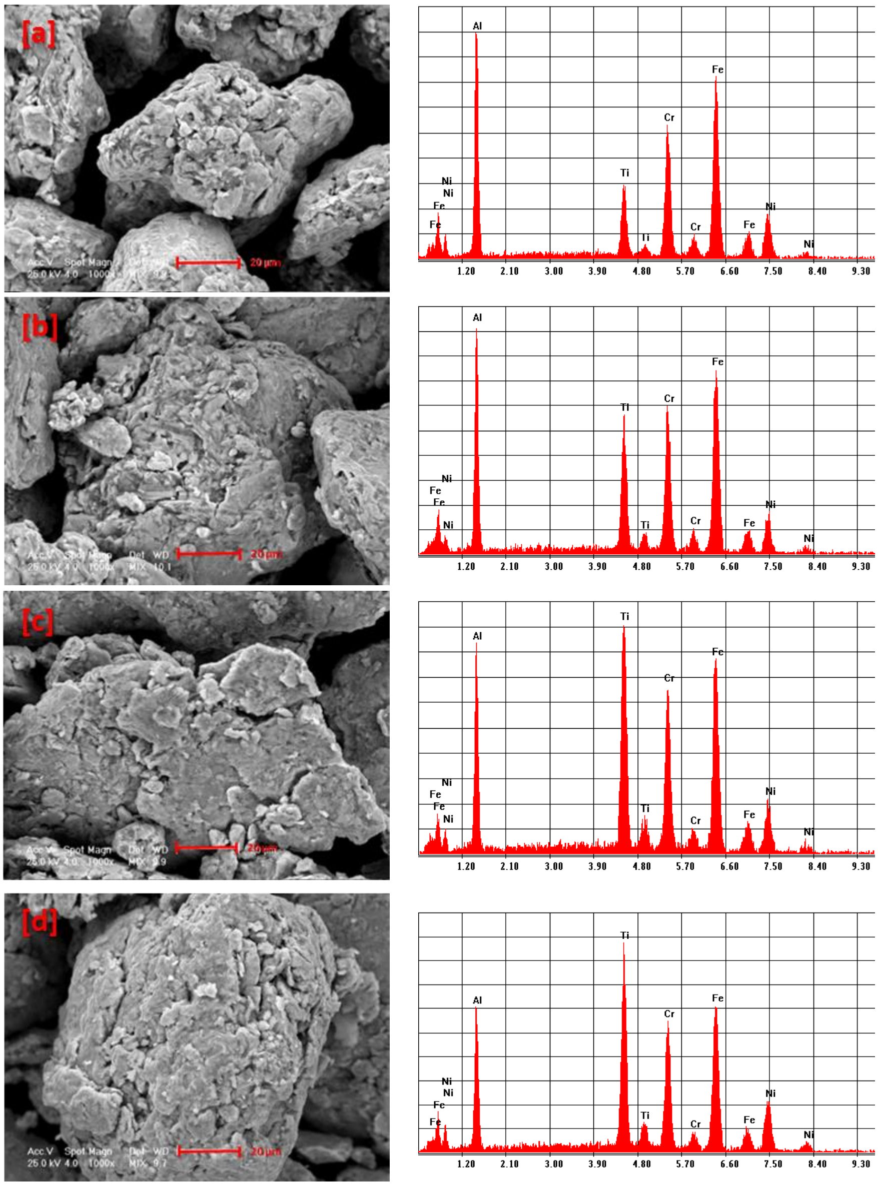

Fig.2. SEM and EDS analyses results for a) 5 h, b)10 h, c) 15 h, d)20 h milling time of AlCrFeNiTi high entropy alloy

It can be observed that after 20h of milling time, the microstructure is very similar with the complete alloy, presented in Fig. 3. The alloying process can be stopped after the 20h due to the fact that after mechanical alloying the powder is subjected to consolidating by pressing and sintering and the alloying process continues during these stages also.

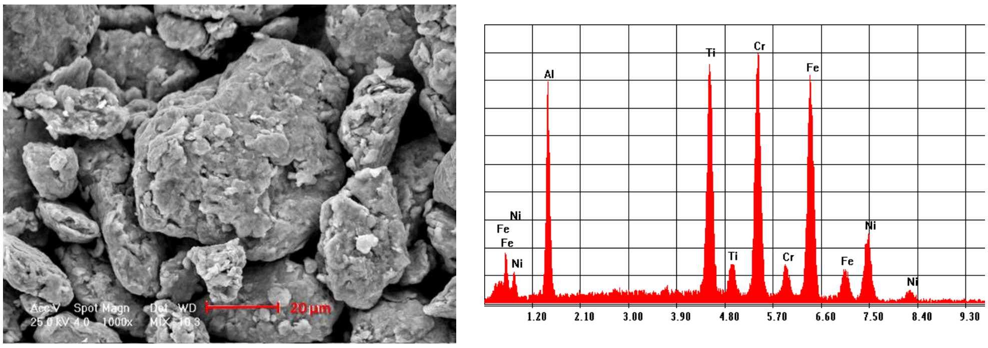

Fig.3. SEM and EDS analyses results for AlCrFeNiTi high entropy alloy after 30 h of milling

The microstructure revealed the homogeneity of the alloyed sample. EDS analyses results show that all the component elements are there. Due to the large milling time and high effectiveness of the ball mill used, the dimension of the obtained particles was reduced considerable and the particles agglomerated very fast according to SEM analyses. From the EDS analyses results can also be observed that in the final alloy there are no traces of contamination.

After obtaining the final alloy, the powder was technological analyzed. Using a Carney funnel, we determined that the Slope angle value for AlCrFeNiTi HEA is of 22.29 degrees. A good flowing of the powder is found to be at angles below 35 degree (Fig. 4).

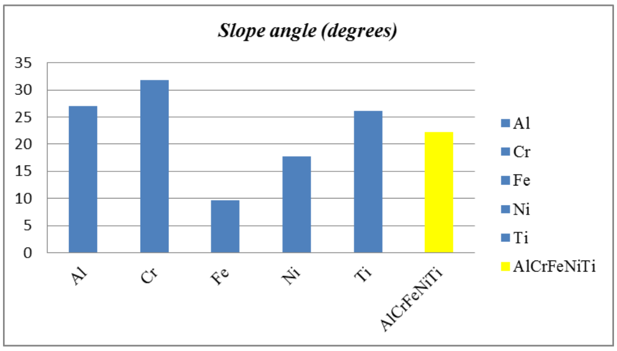

Fig.4. Slope angle value for the raw materials and the final alloy comparison

The graphics (Fig. 5) present the difference between the elemental powders and the final alloy. The free flow density obtained value is of 2.5 g/cm3. The tap density of the high entropy alloy obtained is of 2.98 g/cm3. The packing density represents the ratio between free flow density and tap density, indicating the ability of the powder to be further process by pressing and sintering and for our case the value of 84% shows promising results.

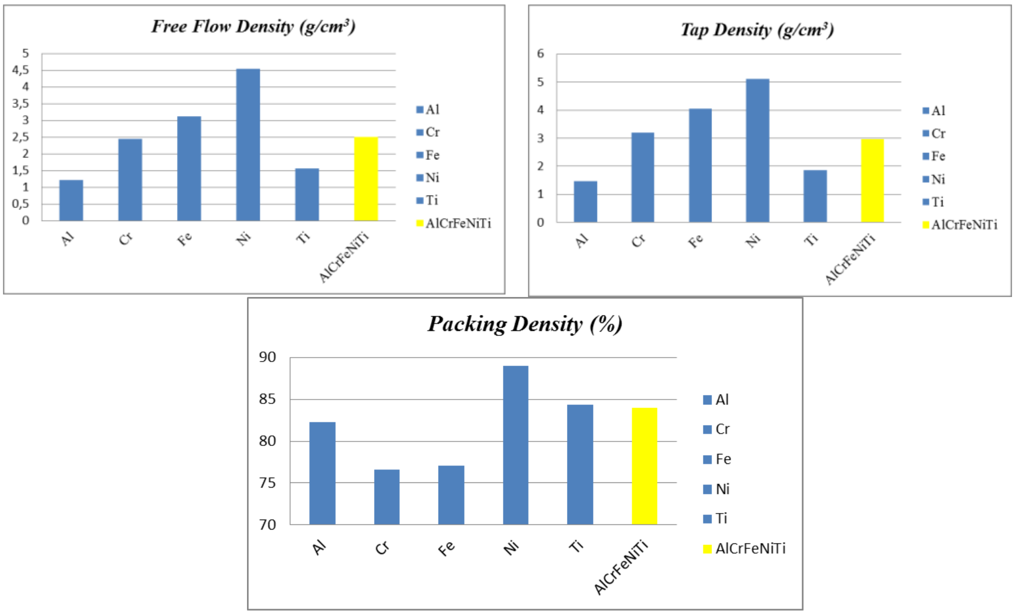

Fig.5. Graphic comparison between the raw powders and the final alloy for free flow density, tap density and packing density

The flow rate value (Fig. 6) of 6.9 g/s indicate that the powder can be deposited by spraying methods as high velocity oxygen fuel, where the risk of clogging the pistol is very high. By comparing the alloy flow with the elemental powders flow can be observed a noticeable improvement.

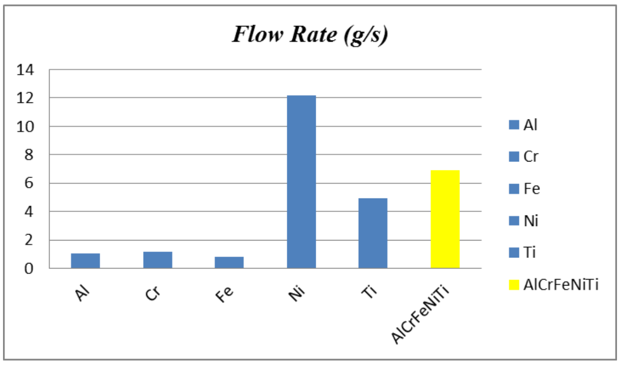

Fig.6. Graphic comparison of the flow rate for raw powders and the final alloy

Due to the spherical shape of the Nickel powder, a better flow rate value was determined.

After the powder characterization, the next step was pressing it into compact samples. Due to the aluminum content, no bonding agent was needed in order to consolidate the sample before the sintering process. The compressibility curves are presented in Fig. 7.

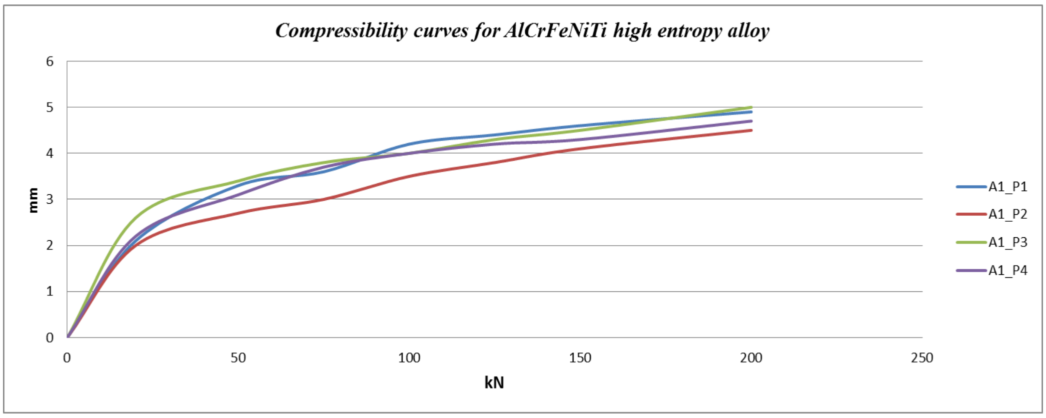

Fig.7. Graphic representation of the compressibility curves for the AlCrFeNiTi high entropy alloy

Multiple tests were performed and for a load of 20 tones force resulting a sample that is compact and can be handled without any difficulty.

After the pressing, the samples were sintered following the sintering curve presented in Fig. 8. The sintering process was conducted under Argon atmosphere, in order to reduce oxidation.

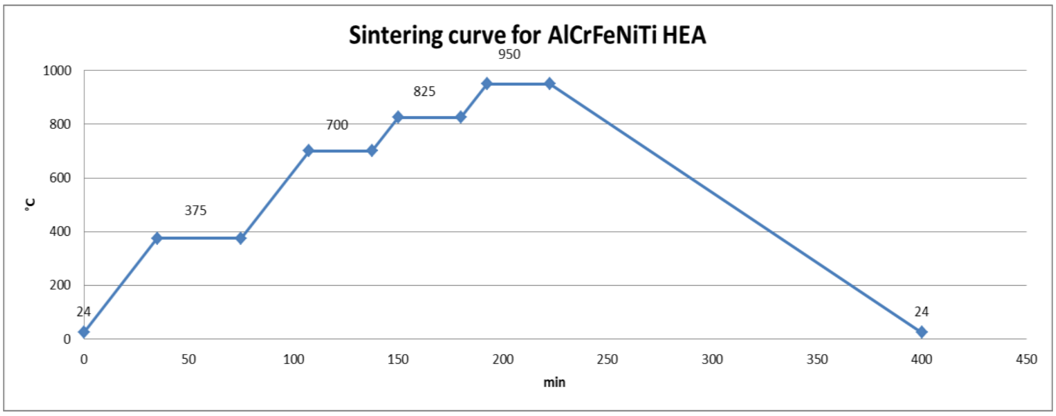

Fig.8. Sintering curve for AlCrFeNiTi high entropy alloy

The samples were prepared metallographic by polishing and cleaning with high purity alcohol, prior to the microstructural analyses that are presented in Fig. 9.

The sintered samples revealed a relatively homogeneity that ensure us the necessary quality for preparing the electrodes that will be further used for coating parts that could be protected against corrosion in various environments. We developed an alloy to be further used for coatings with corrosion resistance. Combining Al and Ti in the same high entropy alloy was proved to be efficient for increasing corrosion resistance as well as workability of high entropy alloys [12].

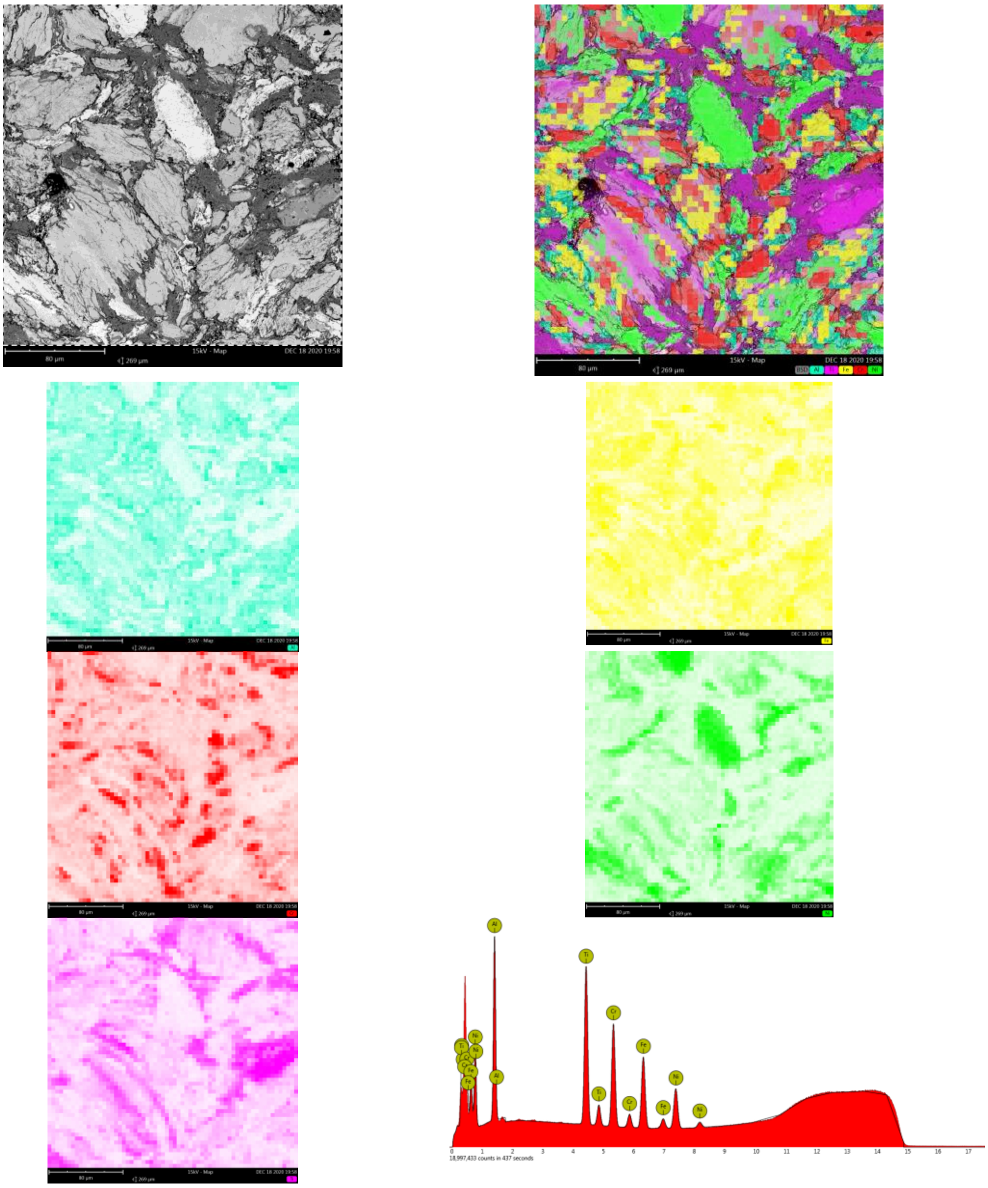

Fig. 9. Microstructure and mapping of the AlCrFeNiTi sintered powder; EDS results of the analysed sample

Fig. 9 describes the microstructure, mapping and EDS analyses of the AlCrFeNiTi high entropy alloy produced by mechanical alloying. The electrodes produced by pressing and sintering are again subjected to temperatures during coating process so we need a lower degree of homogeneity. The mapping confirms that we obtained the alloy with all the elements and the EDS analyses results revealed the elements in the high entropy alloy.

4. Conclusions

In conclusion, the AlCrFeNiTi high entropy alloy produced by solid state processing method was obtained and the ESD and SEM analyses results are presented. A high alloying degree and good homogenization in the final powder was observed.

After obtaining the HEA the powder was metallurgical characterized in terms of packing density, tap angle and flow rate. The values obtained for AlCrFeNiTi high entropy alloy are encouraging for further processing of this alloy.

The powder was pressed, and a compact sample was obtained.

Future work will consist in sintering the compact sample, which will be further machined into electrodes, for producing–coatings of AlCrFeNiTi high entropy alloy by electro spark deposition, a technique suitable for different aggressive environments.

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