Entropic Alloys for Cryogenic Applications

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Abstract

The entropic alloys can be categorized into four types of alloys, e.g., high-entropy alloys, medium-entropy alloys, low-entropy alloys, and pure metals. The high-entropy alloys are a new kind of materials where the mixing entropy plays an important role in the phase formation. Because of the unique structures, the entropic alloys exhibit many outstanding properties, which even break the performance limits of traditional materials, including the excellent low-temperature properties. The mechanical properties of the entropic alloys serving at low temperature are mainly introduced in this chapter, including strength, plasticity, fracture behaviors, and impact resistance, and the reasons for these behaviors reported in recent years are also summed up.

Keywords: entropic alloys, low-temperature brittleness, nano-twins, phase transformation, cryogenic applications

1. Introduction

Materials usually exist in the lowest energy state. It has been clarified in the thermodynamic equation G = H − TS (where G is Gibbs free energy, H is the enthalpy and S is the entropy of the system) that, to reduce the total energy of the system, we can increase T or S, or decrease H, corresponding to the superalloys, high entropy alloys and intermetallics respectively. As a result, the entropy plays an important role in the alloy system, which can be used to define the alloys.

The entropic alloys can be a new categorizing of the alloys filed, which mainly concern the configuration of the components in the alloy systems. The configurational entropy of the alloy can use the Boltzmann equation:

$$\Delta S = kLnW \tag{1}$$

Here, k is Boltzmann constant and W is the number of real microstates corresponding to the macrostate.

The entropic alloys can be categorized into four types of alloys as in **Figure 1**:

1. High-entropy alloys: The configurational entropy ΔS is above 1.61R; here R is gas constant.
2. Medium-entropy alloys: $0.69R < \Delta S < 1.61R$.
3. Low-entropy alloys: $\Delta S < 0.69R$.
4. Ultrapure materials: $\Delta S = 0$.

The high-entropy alloys are a new type of multicomponent materials, which were first proposed by Professor J. W. Yeh of National Tsing Hua University in 2004 [1]. Traditional alloys are usually based on one or two principal elements (usually having a principal content greater than 50%), with the alloy design range locating on the edge of the phase diagram. However, it is broken by the appearance of entropic alloys, and the alloy design range extends to the center of the phase diagram successfully. The entropic alloys usually consist of five or more components, and the atomic percentage of each component is 5–35% with no difference between the solute and solvent [2, 3]. According to the traditional physical metallurgy principle and the Gibbs phase law [4], the more the number of components containing in the alloy, the more likely it is to have a variety of solid solutions and intermetallic compounds. However, in the entropic alloys, the large mixing entropy plays a dominant role to expand the dissolution range of the intermetallic compound and the terminal solid solution, and instead a relatively simple phase structures of face-centered cubic (FCC), body-centered cubic (BCC), or close-packed hexagonal (CPH) are obtained [5–7]. As reported in Yeh's work, when the number of components of the Cu-Ni-Al-Co-Cr-Fe-Si alloy system is increased to 7, only FCC and BCC

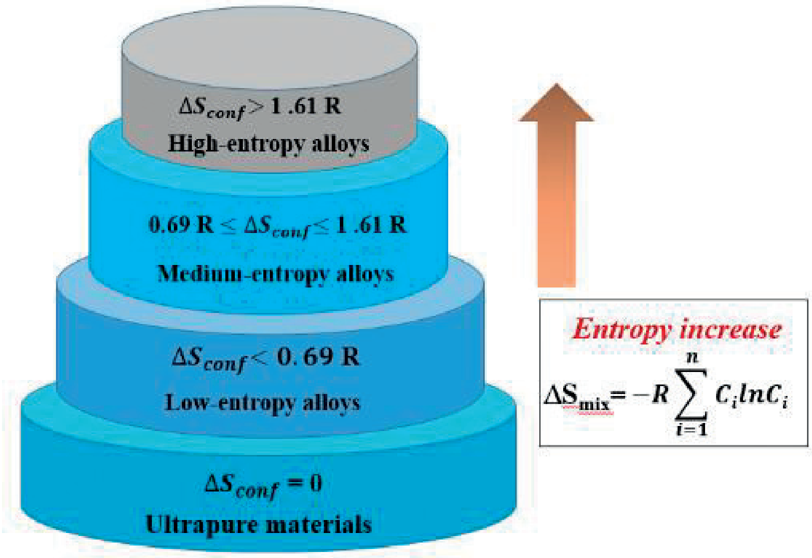

Figure 1. The classification of entropic materials.

simple solid solution structures are still formed [8]. Based on the important role the mixing entropy played in the alloy, it is widely accepted that a molten alloy system where the effect of entropy plays the dominant role ($\Omega \geq 1.1$) and which can form the simple solid solution can be called the entropic alloys [9].

There are a large number of metallic and nonmetallic elements that can be involved in the entropic alloys, including some main group elements in the IIA, IIIA, and IVA group (such as B, Al, Si, Mg, etc.), all the subgroup elements in the fourth period (such as Ti, Cr, Fe, Mn, Co, Ni etc.), and some subgroup elements in the fifth period (such as Zr, Mo, Nb, etc.). Furthermore, there are four core effects in the entropic alloys, including high-entropy effect in thermodynamics, late diffusion effect in dynamics, lattice distortion effect in structure, and cocktail effect in performance. Such a variety of elemental types and the four core effects result in a variety of alloy systems with different properties, which even break the limits of traditional materials, such as high strength [10], high hardness [11], high radiation resistance [12], high temperature softening resistance [13], high wear resistance [14], fatigue resistance [15], etc.

With the rapid development of technology, low-temperature materials are increasingly used in aerospaces, superconducting fields, and civil industries. These kinds of materials can be used in a wide temperature range from below room temperature to absolute zero. It is well known to us that the strength tends to increase while the plasticity and toughness significantly decrease when the conventional materials serve at the low temperature, which in turn results in the cold-brittle fracture. This cold-brittle fracture is similar to the brittle fracture at normal temperature. There is no obvious plastic deformation before fracture, and it suddenly occurs. The crack usually originates from defects or stress concentrations in the material structures and rapidly expands. The type becomes transgranular cleavage, and the fracture feature changes from fibrous to crystalline. This cold brittleness is related to the lattice type of the material. Generally, the BCC and HCP structures are cold-brittle materials, while the FCC metals are non-cold-brittle materials. For the entropic alloys with many outstanding properties and with great application potential in many fields, the researches on low-temperature performance are also in full swing. Therefore, the research status and application prospects of low-temperature properties of the entropic alloys are summarized in this paper, mainly aimed at mechanical properties including strength and fracture behavior.

2. The research status of low-temperature mechanical properties

2.1. The strength and plasticity at low temperature

The most important requirement for materials that serves at low temperatures is to have sufficient strength and stiffness, and in the view of minimizing the total equipment weight, it is often desirable to have the yield strength as high as possible.

There have been a large number of reports about the excellent room temperature tensile properties of the entropic alloys. The crystal structures of the entropic alloys have a significant influence in the tensile properties. Alloys with BCC structures usually have high strength and low plasticity, while alloys with FCC structures are usually accompanied by the large plastic toughness and low strength. However, as the entropic alloys with the BCC structures usually turn to extreme brittleness at the low temperature, researches are mainly aiming at BCC entropic alloys. The earliest research on the low-temperature tensile properties should belong to the E.P. George group of Oak Ridge National Laboratory [16, 17]. Starting from 2013, they systematically studied the low-temperature tensile properties of the FCC entropic alloys and found that the FCC CoCrFeNiMn alloys are characterized by stronger with lower temperature in a wide temperature range. **Figure 2** shows the strength-temperature curves of CoCrFeNiMn and CoCrFeNi alloys at two different strain rates. It can be seen that the yield strengths of these two kinds of alloys have higher temperature dependence and lower strain-rate dependence, and the temperature dependence becomes stronger at lower temperatures (−200–0°C) and higher temperatures (600–1000°C). As the temperature decreases, the yield strength of both alloys increases rapidly, while the elongation does not decrease as the conventional alloy, as shown in **Figure 3**. This abnormal increase of both strength and ductility contributes to the deformation mechanism transforming from dislocation slip to nano-twins, which will be explained later in Section 3.

2.2. The fracture behavior at low temperature

Fracture toughness characterizes the ability of an alloy to resist crack propagation and is a quantitative indicator of the toughness. When the crack size is constant, the larger the fracture toughness value of an alloy, the greater the critical stress required for the crack instability and expansion. When the external force is given, if the fracture toughness value is higher, the critical size of the crack to reach the instability expansion is larger. The fracture toughness can be used as a supplement to the materials which are designed according to yield strength

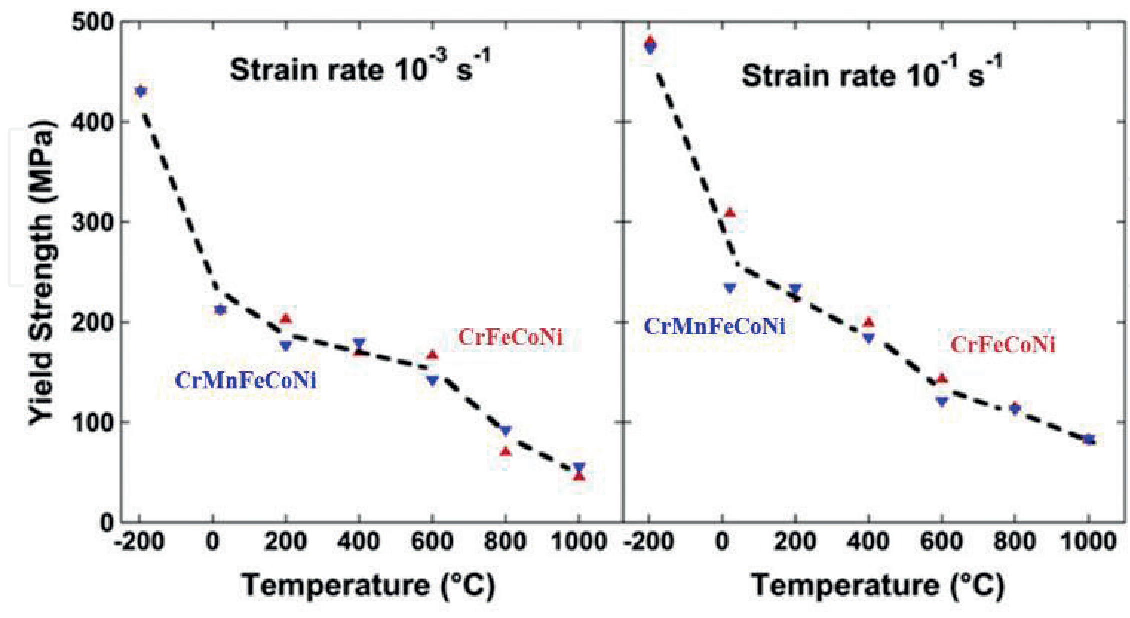

Figure 2. The strength-temperature curves of CoCrFeNiMn and CoCrFeNi alloys at two different strain rates [16].

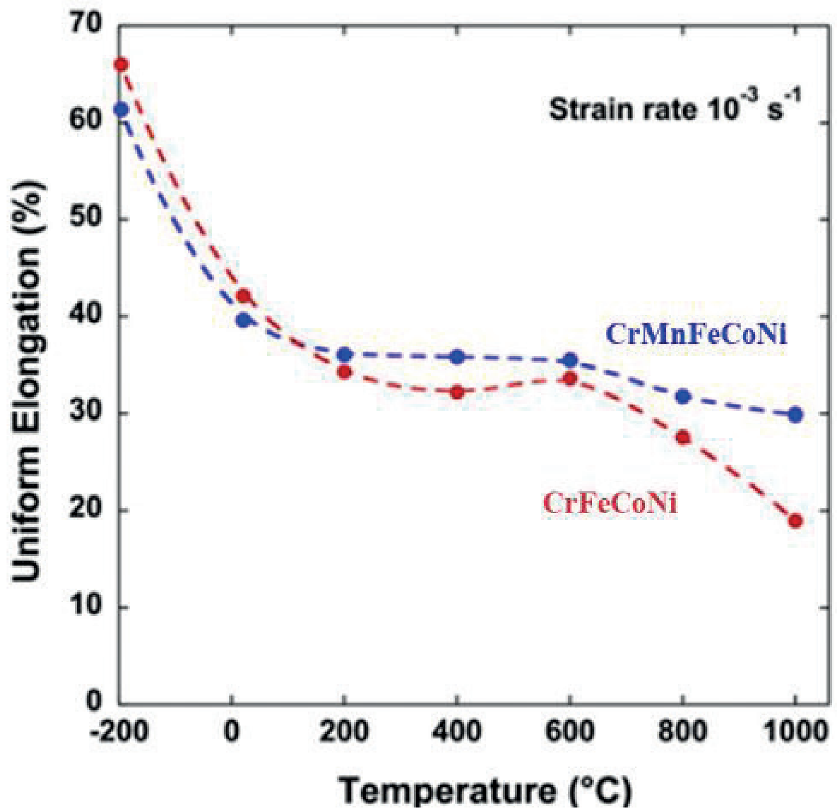

Figure 3. Temperature dependence of the tensile ductilities of CrMnFeCoNi and CrFeCoNi tested at an engineering strain rate of 10^{-3} s^{-1} [16].

criteria. For example, the fracture toughness values usually decrease with the increasing yield strength. Therefore, although the structure of the high-strength alloys has been subjected to a very safe design according to the yield strength criterion, the unstable fracture and fatal damage may occur simply because of an inner crack. Furthermore, since the yield strength of the material increases with decreasing temperature, the low-temperature fracture toughness is not considered in the design process, and the same result may also be caused. Therefore, the characterization of the fracture toughness of entropic alloys is also important.

Professor R.O. Ritchie from the Lawrence Berkeley National Laboratory found the extremely high fracture toughness of FCC entropic alloys [18]. The related research work was published on Science, which arouse widespread concerns of the entropic alloys. It is reported that the CoCrFeMnNi alloys, which are rolled and completely recrystallized, have a tensile strength of 1280 MPa at 77 K, which is 70% higher than that at room temperature. More importantly, its plasticity has been significantly improved, which has fracture strain value greater than 70% at 77 K, 25% higher than that of room temperature, and the work hardening index is as high as 0.4. It also has good fracture toughness. As shown in **Figure 4(a)**, as the temperature increases from 77 to 200 K and then to 293 K, the crack-initiation fracture toughness of CrMnFeCoNi entropic alloys increases slightly from 219 to 221 MPa $m^{1/2}$ and then slightly reduced to 217 MPa $m^{1/2}$. After crack initiation, the fracture toughness of the alloy increases rapidly to 307 MPa $m^{1/2}$ with the growth of a large number of subcritical

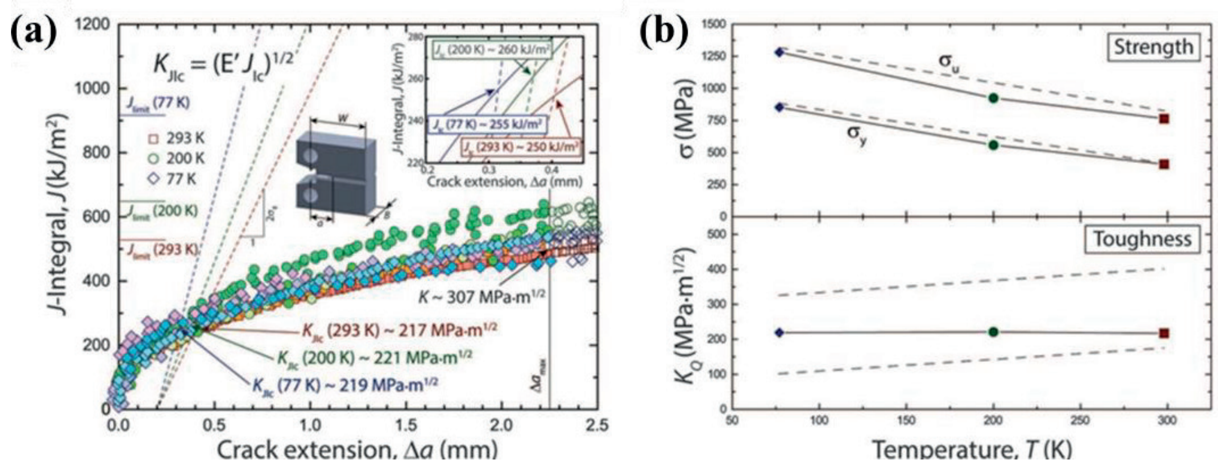

Figure 4. (a) The fracture toughness values change with temperature; (b) the strength and toughness change with temperature [18].

cracks. This high toughness value of entropic alloys can be compared to many high-alloying austenitic stainless steels, such as 304 and 316 L, which has toughness range as K_Q = 175–400 MPa m$^{1/2}$ at room temperature, as well as the best low-temperature steels, which has K_Q = 100–325 MPa m$^{1/2}$ at 77 K. As shown in **Figure 4(b)**, these materials are similar to entropic alloys, and their strengths increase with decreasing temperature. In terms of fracture toughness trends, the toughness of entropic alloys does not change much with temperature, while that of the steels decrease with decreasing temperature.

Subsequent research of this group further found that CoCrNi alloy with equal atomic ratio even has better mechanical properties than CoCrFeMnNi in low-temperature environment, and the fracture toughness at 77 K can reach 273 MPa m$^{1/2}$ [19], which indicates that it is the nature of elements in complex solid solutions rather than their mere numbers that is more important. Indeed, in terms of (valid) crack-initiation and crack-growth toughnesses, the CrCoNi medium-entropy alloy represents one of the toughest materials in any material class ever reported, as manifested in **Figure 5**.

2.3. Impact resistance at low temperature

When the materials are applied at low temperatures, they will be subjected not only to conventional tensile/compression loads but also to the impact loads. Therefore, it is necessary to test the impact toughness by using a pendulum test. The higher the impact toughness is, the more impact energy is absorbed, indicating that the material's ability to withstand impact loads is better.

Utilizing the Charpy impact test standardized in the ASTM standard E-23, Li et al. found that the homogeneous structures of the $Al_{0.1}$CoCrFeNi and $Al_{0.3}$CoCrFeNi entropic alloys after hot forging are obtained, the casting defects are reduced, and the excellent tensile properties and impact resistance are exhibited at low temperatures [20]. The V-notch impact test at 77–298 K shows that the Charpy impact energy of $Al_{0.1}$CoCrFeNi alloy decreases from 420 to 289 J with the decrease of temperature, while it reduces from 413 to 328 J in $Al_{0.3}$CoCrFeNi, which is significantly better than traditional materials such as stainless steels and titanium alloys (as shown in **Figure 6**).

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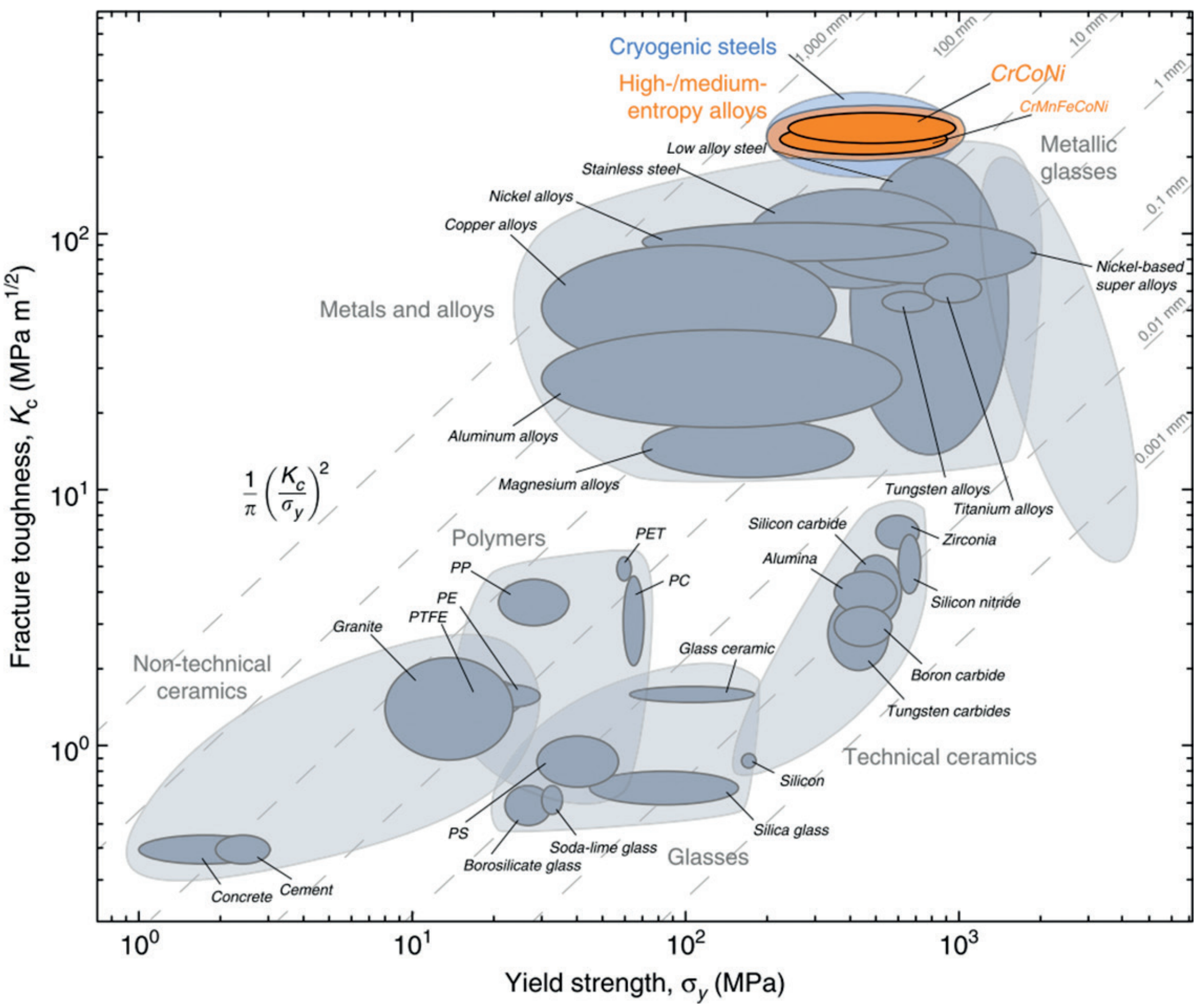

Figure 5. Map of fracture toughness versus yield strength for various classes of materials [19].

However, the Charpy impact test did not exhibit the same increase in strength and plasticity at low temperatures just as the tensile test, which may be owing to the sharp increase in the energy required for dislocation slip under high-speed deformation conditions.

In the impact performance experiment, the type of notch is closely related to the impact toughness. Different types of notch make the stress concentration different. For example, the V-notch reflects the crack propagation, while the U-shaped gap reflects the initiation. At the same time, the state of the material also affects the impact properties. The impact resistance of the Al_xCoCrFeNi after hot forging is reported by Li et al., and the researches on the as-cast Al_xCoCrFeNi (x = 0, 0.1, 0.75, 1, 5) alloys are carried by Xia et al. [21]. As the Al content increases, the alloy changes from a FCC structure (Al_0 and $Al_{0.1}$) to a BCC structure ($Al_{0.75}$ and Al_1), which then bring huge changes to the impact toughness of the alloys. The impact toughness of the as-cast cubic Al_0 and $Al_{0.1}$ alloys at low temperatures is slightly poorer than that of hot-forged alloys [20], but it still has excellent impact resistance, and the impact energy increases with temperature decrease, which have opposite tendency from that in the hot-forged alloys. The impact energy of $Al_{0.1}$ at 77 K is slightly lower than that of Al_0, but it becomes slightly higher as temperature increases.

The impact energy of Al_0 at 77 K is 398 J, which is the best in all alloys. And the average impact energy of the V-notch sample is slightly higher than that of the U-notch sample. It can be seen from the morphology of the sample after the impact tests that Al_0 and $Al_{0.1}$ are destroyed from the notch without broken into two pieces, while the other two alloys were completely impact broken, indicating that their impact resistance are absolutely poor (**Figure 7(a)** and **(b)**).

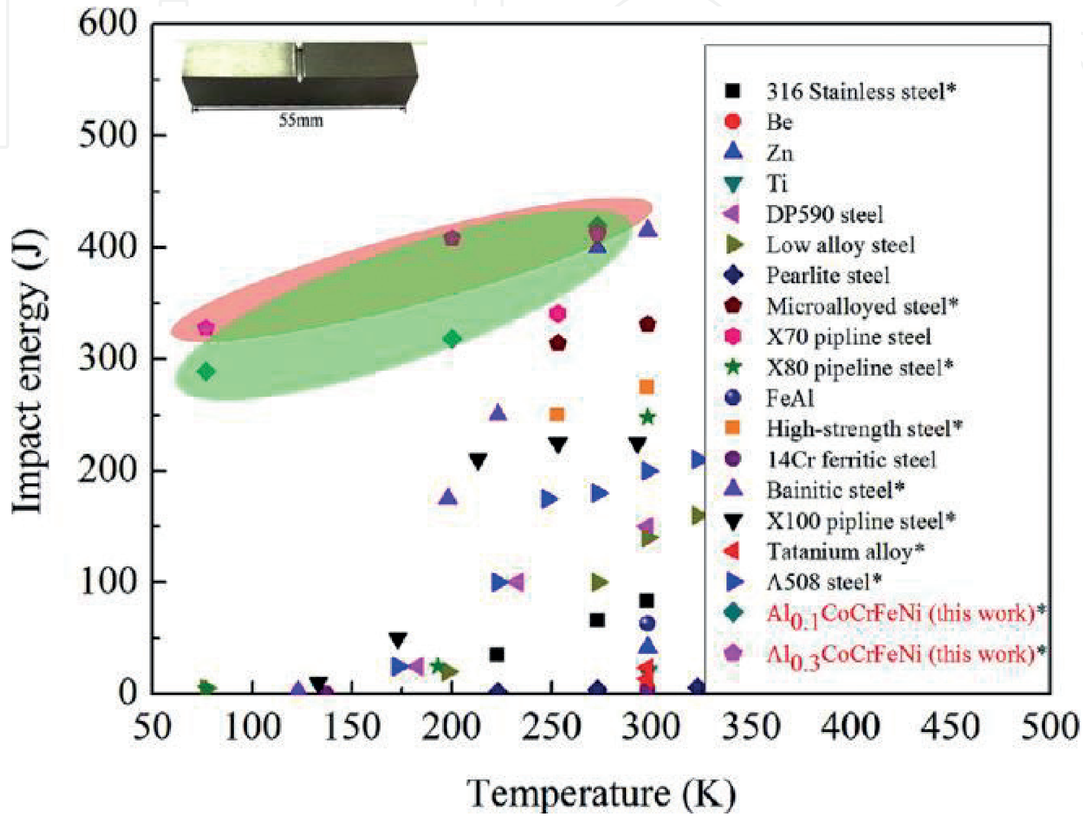

Figure 6. Summary of the Charpy impact energy of materials at the different temperatures [20].

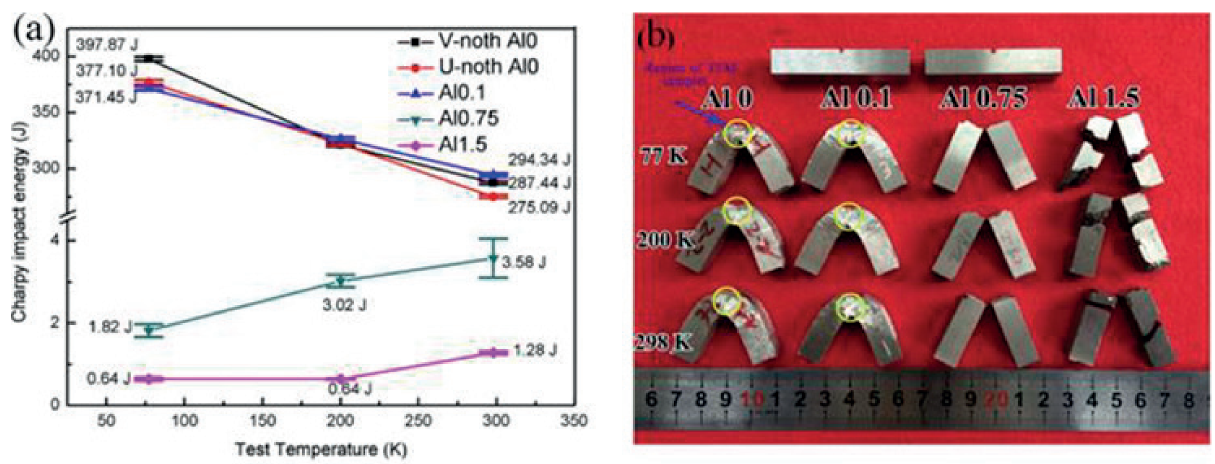

Figure 7. (a) The impact energy as functions of different test temperatures and aluminum contents; (b) the macroscopic samples after impact [21].

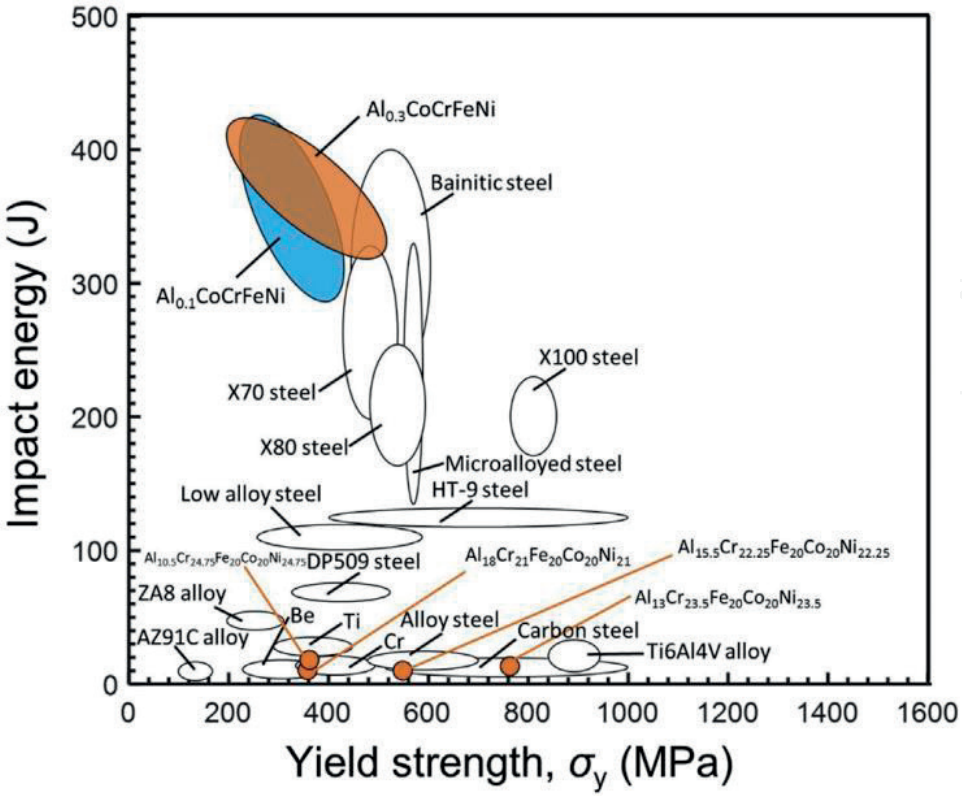

Figure 8. Map of impact energy versus yield strength for different materials [25].

In all these alloys, just as **Figures 6** and **7(a)** show, no obvious ductile-to-brittle transition (DBT) occurs as in many such conventional alloys as steels, amorphous alloys, Mg alloys, porous metals, and nanocrystalline metals [22–24]. A great DBT property and a low DBT temperature are detrimental to cold condition applications. Nowadays, researchers have tried to reduce the DBT temperature of materials through various methods, and the emergence of these kinds of entropic alloys provides them with a good choice.

Furthermore, it can be seen from the impact energy-yield strength diagram shown in **Figure 8** [25] that the two kinds of entropic alloys $Al_{0.1}$ and $Al_{0.3}$ have excellent comprehensive mechanical properties of impact energy and yield strength. Although their yield strength is relatively smaller than that of some carbon steels, their impact toughness is several times to several tens of times higher than that of various steels and titanium alloys.

3. Deformation mechanism at low temperature

3.1. Synergistic effect of dislocations and nano-twins

Stacking fault is a lattice defect that only changes the sub-neighbor relation of atoms and hardly produces lattice distortion. Usually, the energy added by the unit area fault is called the stacking-fault energy, which is an intrinsic property of metals. The smaller the stacking-fault energy, the greater the probability of stacking faults occur. In low-stacking-fault-energy alloys, such as austenitic steels and magnesium alloys, dissociation into partial dislocations is more energetically favorable, and the spacing between the partial dislocations is larger. As the spacing increases, cross slip and climb become more difficult, increasing strength. Low-stacking-fault-energy alloys are also more likely to deform by twinning, increasing dislocation storage capacity, strain-hardening rate, and ductility.

Huang et al. found that the entropic alloys have lower-stacking-fault energy and are particularly effective in low-temperature environments [26]. Based on this, it can be inferred that the entropic alloys will undergo twinning at low temperatures and have excellent performance, which has been confirmed by the subsequent studies.

A large number of experiments have studied the nature of the excellent low-temperature properties of entropic alloys from various aspects. It is now believed that the synergistic effect of various deformation mechanisms and the inhibition of crack growth by nano-twins are the essence of excellent mechanical properties [27]. In the room temperature, the deformation is mainly realized through dislocation, and the proliferation and expansion of large numbers of dislocations make the entropic alloys have excellent plasticity. The dimple diameter is almost entirely micron size (1–10 μm). While in the low temperature, nano-twins appear and interact with dislocations. Among different grains, the twin exhibit strong synergistic deformation, while a large number of cross twisting appear in the same grain. The interaction of the oriented twins greatly refines the grains, and the nanoscale dimples appear in the fracture. The combination of these factors makes the entropic alloy exhibit excellent low-temperature performance.

3.2. Phase transition

Further in-depth studies have found that there are small amounts of phase changes in the deformed sample, that is, the FCC to HCP phase transition occurs when the material is deformed in the liquid nitrogen and liquid helium temperature zones. In the ultralow temperature, the FCC to HCP transition fully demonstrates that HCP is a stable phase at low temperature and the FCC structure is a metastable phase, which is similar to the deformation behavior of 316LN in ultralow temperature. However, differences are that the volume fraction of 316LN in the phase transition is about 24%, while the entropic alloys only have a very small amount of FCC phase transformed. This also indirectly indicates that the entropic alloys have a more stable FCC structure than the conventional materials due to their high mixing entropy.

The results of TEM and HRTEM show that the dislocation decomposition produces partial dislocations, and the slip of them in every other layer of the close-packed planes causes the phase transition. Due to the special orientation relationship between FCC and HCP, coupled with the promotion of low temperature, atoms in every other 111 layers which stack in the order of ABCABC form glide with Shockley partial dislocation, which in turn cause the appearance of HCP structure as the stacking order of ABAB.

In addition, although the phase transformation is beneficial to improve the strength and plasticity, the stress concentrations are also prone to occur in the boundary, and the occurrence of twinning hinders the dislocation motion. Therefore, the plasticity is lower than that at 77 K.

4. Applications and prospects

With the continuous development of science and technology, low-temperature technology has played an increasingly important role in human production since its discovery, especially in aerospace science, biology, and life sciences. In the low-temperature state (including extremely low temperature), the properties of the substance may change dramatically: gas liquefaction or solidification, slow or stop of biological cell metabolism to prolong its life, loss of electrical resistance of the conductor to form a superconductor, and the like. Therefore, both the development of refrigeration cryogenic equipment and the application of ultralow-temperature materials have greatly promoted the development of science and technology and greatly improved the level of human production, especially in the treatment of major human diseases and the exploration of outer space, such as the development of LNG cryogenic carriers and low-temperature superconductors. The development of cryogenic engineering relies on the development and renewal of low-temperature materials.

A large number of experimental studies have shown that the entropic alloys have excellent low-temperature mechanical properties. It has been investigated that these great mechanical properties far exceed those of some high-alloying austenitic stainless steel and they have great potentials for low-temperature applications, including low-temperature storage and low-temperature structural parts. The researches on the deformation mechanisms are also becoming more and more mature. However, there are still some problems that need to be resolved. Firstly, the low-temperature performance of entropic alloys in different states, different deformation levels, and different heat treatment processes also requires systematic research. Secondly, when serving at low temperatures, not only excellent mechanical properties are required, but also corrosion resistance and easy processing ability are needed for the alloys, which still need to be further investigated. Furthermore, another interesting phenomenon is that the stress-strain curve appears to be serrated in a specific temperature and strain-rate range [28, 29], which will seriously affect the properties and applications, because the unsmooth stress-strain curve is difficult to predict the material safety factor. As a result, it is necessary to study the serration behaviors more thoroughly.

5. Conclusion

The entropic alloys have excellent low-temperature mechanical properties, including high tensile strength, high fracture toughness, and high impact resistance, some of which even exceeds the limits of existing materials. The main reason for its excellent performance is that the ultralow-temperature environment and high-entropy effect are beneficial to reduce the stacking-fault energy, which makes it easy of deformation mechanism transition from dislocation dominant to twining dominant. The appearance of cross twisting and synergistic twinning greatly improves the low-temperature properties. In addition, the local FCC to HCP phase transitions also affect their comprehensive properties.

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Conflict of interest

The authors declare that they have no conflict of interest.

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