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High-Entropy Alloys for Micro- and Nanojoining Applications

Ashutosh Sharma

Abstract

The aim of this chapter is to provide a basic understanding of the metal-ceramic joints and high-entropy alloy (HEA) research in microjoining applications. We will first overview the issues in metal-ceramic brazing and solutions to overcome those issues using various fillers. Various approaches are available for joining ceramic to metallic materials. One approach will be to look for brazing alloys with the so-called high-entropy characteristics which exhibit a solid solution phase. The conventional alloy design and arc melting, Bridgman solidification, and advanced powder metallurgy techniques will be studied, including high-energy ball milling (HEBM) for the mechanical alloying process, and hot-press and spark plasma sintering (SPS) techniques are utilized for improved densification and phase transformation. We also summarize the various thermodynamic relations to obtain the high-entropy phase and present future possibilities of high-entropy alloys in microjoining research at the later stage of this chapter.

Keywords: brazing, entropy, microjoining, welding, alloy, wetting, strength

1. Introduction

There are various types of technologies to bond metal-metal, metal-ceramic, and ceramic-ceramic couple. To achieve this we need a bonding agent known as filler or brazing alloy [1]. The most popular one is active metal brazing where the filler consists of a reactive element, like Ti, Zr, Nb, etc. Another approach is to coat the ceramic body with an active Ti layer. However, to utilize the maximum benefit of the active element, additional surface-active material is required to prevent the generation of residual stresses [2]. Also, the unwanted reaction products by the addition of a number of elements in the filler alloy further deteriorate the joint by creating additional intermetallic compounds (IMCs) as crack generation points. Thus, the formation of IMCs also needs to be controlled for better reliability of the joint [3].

At present, several researchers have used brazing of Al_2O_3, ZrO_2, Si_3N_4, SiC, etc. by using active filler brazing. Active metal brazing makes joining of high melting point materials feasible without melting the contact specimens [4, 5]. Various active filler metals, such as Ag-Cu, Ag-Cu-Ti, Ag-Cu-Sn-Ti, Ag-Cu-In-Ti, Ag-Cu-Ti, Ag-Ti, Ag-Zr, Ti-Zr-Ni-Cu, TiNi, etc., have been investigated for brazing of ceramics. The addition of several active elements can also contribute to various IMCs, Cu-Ti, Cu_3Ti, Ti_3SiC_2, etc. Due to these brittle IMCs, the joint cannot be operated at high temperatures [6–12]. Therefore, the key factor to braze ceramics to metals is to look for new fillers for brazing

where active elements (Ti, Zr, etc.) distribute randomly in solid solution. In addition, the alloy system should be free of any additional undesirable IMCs. Recently discovered high-entropy alloys have already shown that with proper design strategy, they might replace traditional materials for a variety of applications [13].

2. Metal-ceramic brazing

In recent microjoining communities, ceramic-metal bonding is of prime importance due to its various applications in hybrid joints, such as ceramic packaging, ceramic sealants, thermoelectrics, and semiconductor transducers. The various types of microjoining processes are given in **Figure 1**.

However, due to the wide limitations of joining this couple owing to their wide gap in physical, mechanical, and thermodynamic behavior, they have always imposed a difficulty. Ceramics are lightweight, harder, and always inert toward the liquid metal and have a wide difference in thermal expansion behavior. Since metals are soft and malleable, for various functional applications, their joining is needed in semiconductors. There are various types of filler materials already available, such as Ag-Cu-Ti brazing system which is most suitable in metal-ceramic joining [7, 14].

The joining processes include various soldering, brazing, and welding processes depending upon the range of temperature of the filler. Soldering is a low-temperature joining but weaker for most of the ceramics due to high-temperature
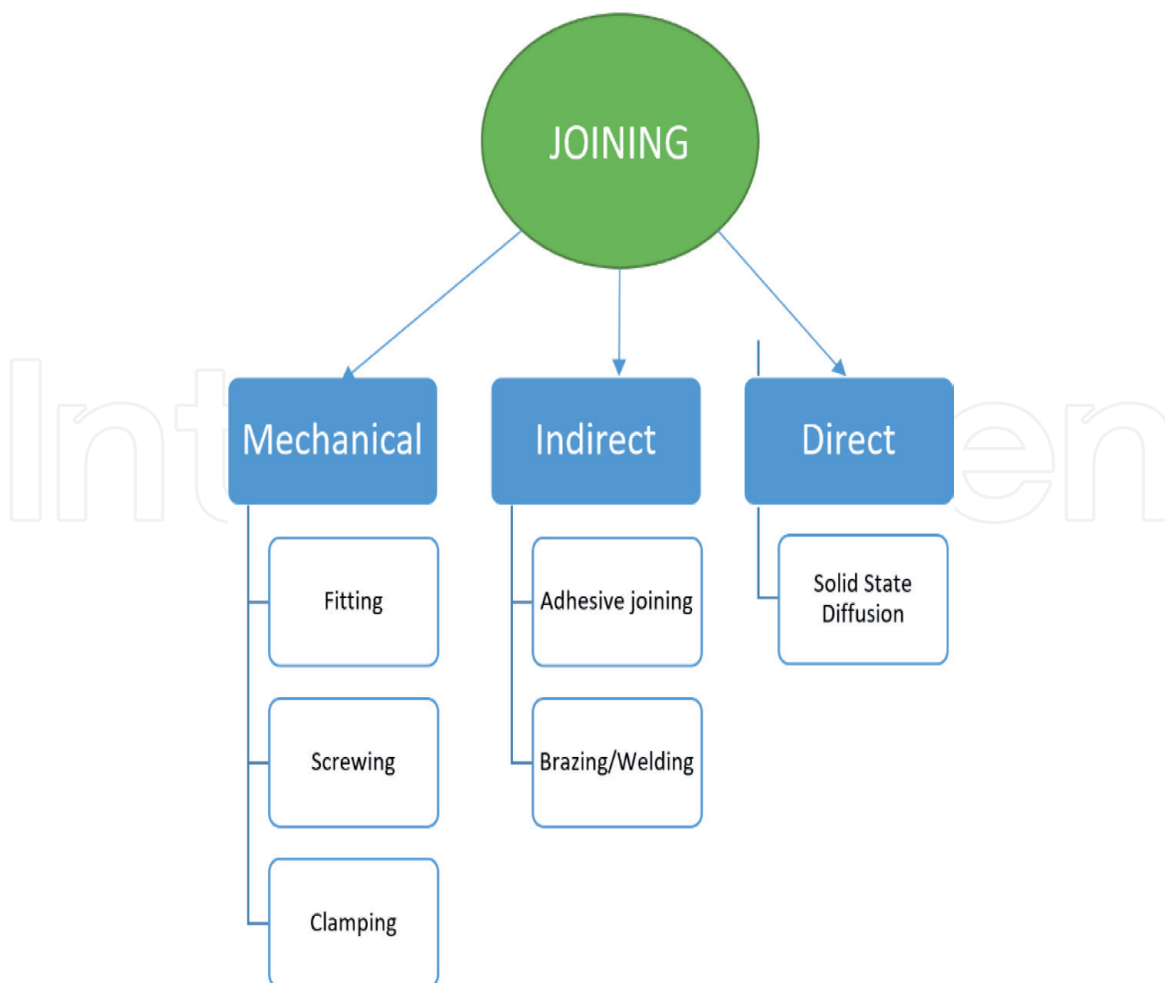

Figure 1.
Various types of metal-ceramic joining processes.

Figure 2.
Various types of welding processes according to the temperature of the application.

requirements. A solder works below 450°C and may not work for high-temperature superalloy joining, while welding is a high-temperature process mostly used for steels [15–18]. In welding, the metal parts need to be melted at high temperatures using fillers known as welding rods which solidify and bond to the workpiece. However, this may not be suitable for the bonding of different metal parts where thermal damage is of concern. A brief detail of the various types of welding processes is given in **Figure 2**.

3. Necessity of developing brazing fillers

Enormous amount of work has been done on the lightweight materials for automotive, defense, and aerospace applications in the last few decades, mainly on steel, ceramics, aluminum, and magnesium-based alloys. However, not all the materials have led to mass production for practical use. The studies reported are preliminary wherein most of the time, microjoining is ignored even if it is an essential part of the automobile, aerospace, consumer, and defense electronic industries.

Therefore, a new structural material, for example, for automobiles, cannot be designed without any foundation for microjoining that is an essential component to maintain the structural integrity of the part in service. There are several important problems in microjoining, such as the presence of brittle IMCs at the interface, segregations, and casting defects (shrinkage porosity, gaseous inclusions, pores, blowholes, etc.). The situation becomes more serious for ceramics (ceramicmetal, ceramic-superalloy, ceramic-steel, etc.) [19]. For example, in various complex assembly applications (ceramic-metal plugs for igniting car engines), ceramics are difficult to join with metals due to their inherent poor wettability and difference in coefficient of thermal expansion (CTE). As shown in **Figure 3**, these two problems have limited the application of ceramics in combination with metals for many years. In this chapter we mainly focus on the brazing which is the most popular for ceramic-metal joints. However, first, we discuss a bit more about brazing processes.
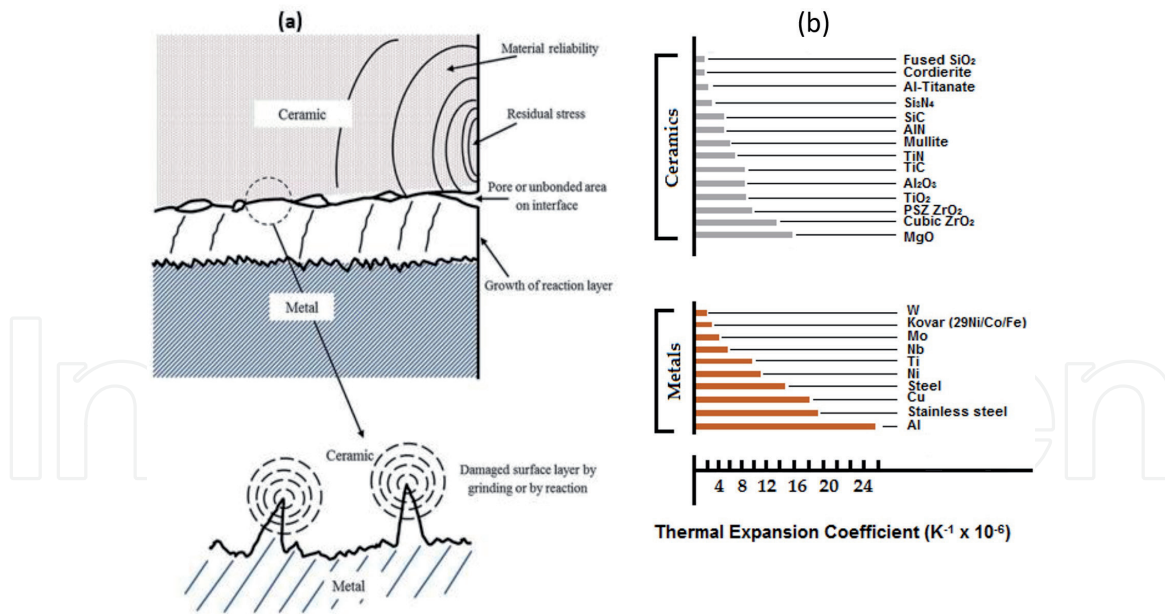
Figure 3.
Metal-ceramic bonding issues. (a) Schematic of factors influencing on the reliability of ceramic-metal joint and (b) comparison of thermal coefficients of metals and ceramics. © 2016 Uday et al. [19] under CC BY 3.0 license.

4. Active metal brazing

There are various brazing processes, such as diffusion bonding, transient liquid phase bonding, reactive bonding, etc., out of which active metal brazing is more popular due to its suitability to ceramics. Active metal brazing enhances the surface wetting of the contacts due to the presence of active elements in the fillers. Recently authors have bonded the ZrO_2 and Ti-6Al-4V using a composite filler of Ag-Cu-Ti to obtain a robust brazed joint [20]. Active metal brazing minimized the residual stresses and distortion in the joint poor strength and high-temperature stability are the issues in brazing industries. To understand brazing, we should have an idea of the wetting of the two contact surfaces. We describe the wetting theory in the following sections.

4.1 Theory of wetting and spreading

According to the wetting theory formulated by Thomas Young in 1805, the interfacial surface energies at the solid–liquid–vapor interface are related by an equation:

$$\gamma_{sv} - \gamma_{sl} = \gamma_{lv} \cos \theta \qquad (1)$$

Here, γ_{sv}, γ_{sl}, and γ_{lv} correspond to the interfacial energies of solid-vapor, solid–liquid and liquid–vapor interface (**Figure 4(a)**). According to the spreading parameter,

$$S = \gamma_{sv} - \gamma_{sl} - \gamma_{lv} \qquad (2)$$

$\gamma_{sv} - \gamma_{sl} - \gamma_{lv} = 0$ for minimum surface energy (high spreading) and $S < 0$ for poor wetting. **Figure 4(b)** gives the wetting angles of different metals at various temperatures [7].

We can see that the temperature has a great role in the wetting of metal fluid on the substrate. The increase of Ti and Sn in optimal proportion leads to an increment
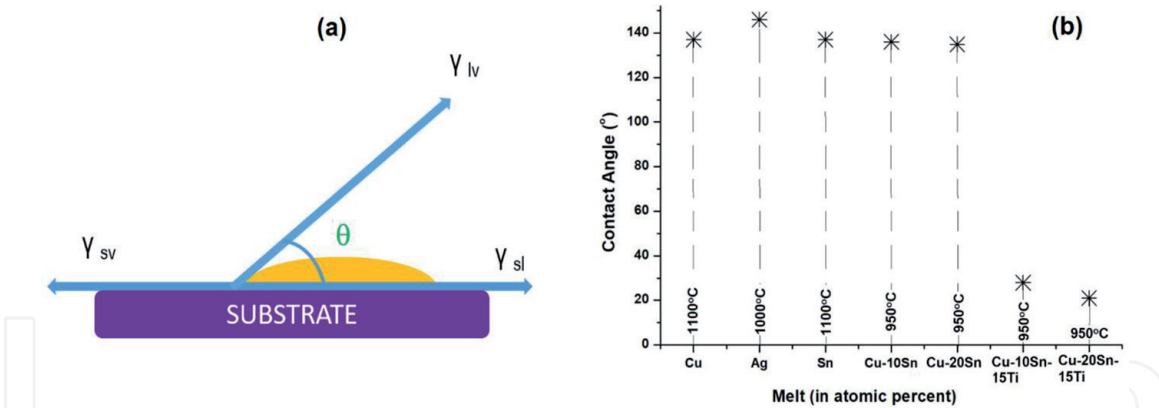
Figure 4.
(a) Schematic of filler wetting at the substrate and (b) contact angle of metals on cBN substrate. [the data in (b) is adapted from [7]].
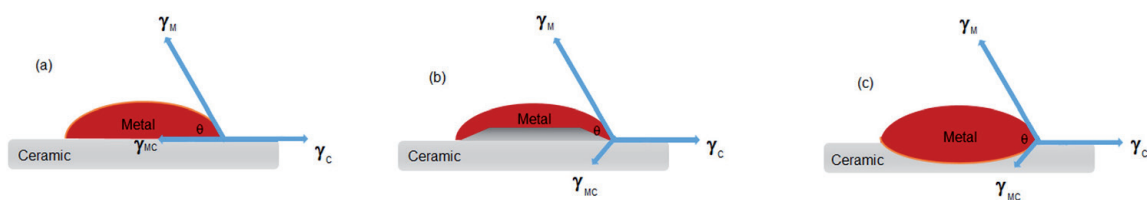

Figure 5.
Liquid metal drop shape depending on the contact time: (a) initial contact or rigid solid surface, (b) formation of a ridge (vertical scale exaggerated), and (c) final equilibrium configuration on deformable solid. © 2016 Uday et al. [19] under CC BY 3.0 license.

in the wettability of the contact surface. The various situations of wetting are given in **Figure 5** depending on the contact angles between the metal and ceramic substrate.

4.2 Factors influencing brazed joint

4.2.1 Issues in metal-ceramic wetting

The wetting is influenced by the surface energy, reactivity of the contact materials to brazing filler, and type of filler used. The reaction between the contact surfaces and brazing filler is key to improve the wetting. In the active metal brazing, a higher wetting can be achieved using Ti, Sc, Cr, or Zr active metals that minimize the surface energy and enhance wetting [21].

4.2.2 Ag-Cu-Ti filler system

Various active fillers are developed in the past. The authors will focus mainly on Ag-Cu-Ti among conventional fillers. This alloy system has various features such as solid solution Ag(Cu) and intermetallic phase Ti_2Cu and Ti_2Ag compounds. The melting range is around 800–900°C and, therefore, is useful to bond ceramics-metals for most of the systems. Ag promotes the activity of Ti and reaction proceeds at a faster rate. There is no ternary intermetallic in Ag-Cu-Ti system [22].

4.2.3 Surface condition

Sharp edges and corners may raise the stress concentration and completely fail the ceramic-metal joint. There should be no surface cracks as they propagate

to form a bigger crack which ultimately leads to joint failure. The damage can be repaired through re-sintering the part [19].

4.2.4 Thermal characteristics

High-temperature gradients may cause the generation of thermal stresses and may not be desirable from the brazing point of view. In ceramic-metal bonding, if the ceramic has less CTE than metal, edge cracks will be formed, while core cracks are formed in the opposite case, as shown in **Figure 6** [19, 23, 24].

The remedy for minimizing these cracks is given by various researchers. Zhou [25] suggested the application of metal layer as an interlayer to release the stress by elastoplastic deformation, using composite layers, low-temperature joining, and proper heat treatment and joint design.

4.3 Interface characteristics

The presence of chemical bonding also affects the brazing performance. For example, a thick interfacial layer causes more mismatch in thermal expansion and weakens the joint compared to a thin layer [26]. The operating temperature and time are key parameters to control the interfacial layer. The presence of pores and edges should be avoided before brazing to control the contact angle of the filler with the substrate.

4.4 Type of filler

Filler type also affects the brazing operation. Researchers have also used nanotechnology to develop various brazing fillers, known as composite fillers [27–31]. These nanocomposite fillers refine the various IMCs present in the matrix and joint. However, the dispersion of nanoparticles in the composite matrix is cumbersome. For example, the active metal filler has been reinforced with metallic or ceramic additives to match the CTE and promote the brazing [20]. In the last few decades, the research community has found the solution to overcome the forma-tion of IMCs in an alloy. Such a novel type of alloy design is known as high-entropy alloys as we describe in the following section.
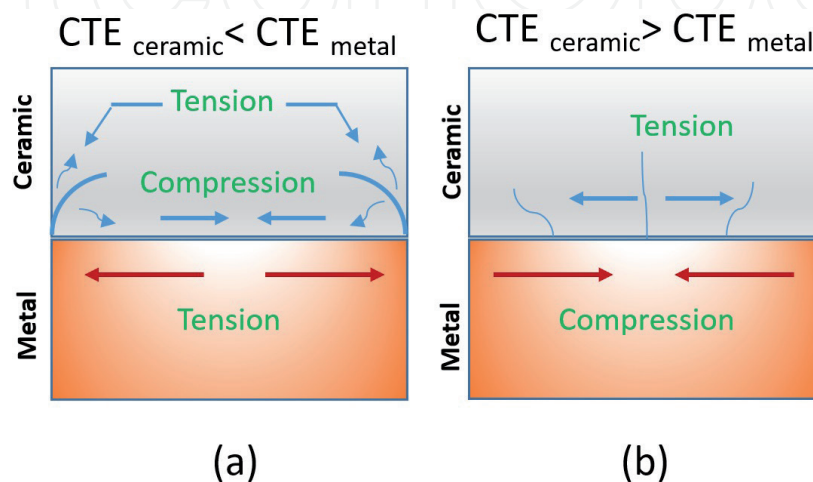
Figure 6.
Types of cracks in metal-ceramic joints. © 2016 Uday et al. [19] under CC BY 3.0 license. (a) Edge cracks in ceramic. (b) Core cracks in ceramic.

5. Advanced brazing fillers must be of high-entropy characteristics

Conventional alloy design suggests the formation of various intermetallic compounds (IMCs) or complex phases with multiple alloying elements. These IMCs act as stress raiser points in alloys and composites which are strictly not desirable. Specially in microjoining applications, these IMCs can cause poor joint properties and catastrophic failure of the entire device. Yeh et al. broke this paradigm by suggesting high-entropy alloys (HEAs), composed of five or more elements in an equiatomic or near-equiatomic fraction varying from 5 to 35 at.% [32, 33]. Therefore, these HEAs contain usually simple solid solution phases rather than IMCs. Many HEAs with high strength, thermal stability, excellent corrosion and wear resistance have been reported. However, they are rarely explored for microjoining applications. Application of these novel HEAs in microjoining can provide several benefits. High entropy filler alloy realizes the active interfacial reaction with ceramic, and the solid solution forms the brazing seam during brazing. The interaction of additional IMCs in the brazing seam is prevented (unlike traditional brazing fillers). This further provides additional benefits like a reduced requirement of ceramic metallization partly with active metal (Ti, Zr), prevents stresses and distortion in joints, improves joint strength, depresses the brazing temperature, and saves energy.

6. Fillers processed by powder technology

Most of the brazing fillers so far available for ceramic-metal joining are silver-based alloys mostly produced by casting approach, e.g., Ag-Cu, Ag-Cu-Ti, Ag-Cu-Sn-Ti, Ag-Cu-In-Ti, etc. [19–26]. Several elements are added to enhance the wetting of the contact surfaces which further produce unwanted IMCs. Moreover, conventional casting or melting techniques are not suitable for industrialization as they produce a number of defects (shrinkage porosity, gaseous inclusions, pores, segregation of constituents, impurities), and there are limitations on the shape/size of the final product. In contrast, the advanced solid-state powder technology has rarely been used for HEA synthesis, and there is a lot more to explore. It offers a low-cost superior alternative over other expensive techniques (sputter deposition, evaporation, induction melting, arc and laser melting, etc.) Secondly, it is an environmentally benign and safe technique as compared to chemical routes (solution precipitation, plating, chemical vapor deposition) where toxic waste disposal and drainage costs are added up [17–19]. In addition, high-energy ball milling is a simple and effective way to produce novel nanostructured materials, homogeneous chemical distribution, and extension of solid solubility and widens the scope of HEA [34–40]. Further densification of milled powders by heat treatment, hotpressing, cold isostatic pressing, or spark plasma sintering (SPS) technique gives the stable bulk HEA.

7. Research and development on HEA in microjoining

HEAs are the novel and advanced alloys which are composed of 5–35 at.% where all the elements serve as principal elements. They are unique because of their attractive properties compared to their conventional alloys such as strength and ductility trade-off, high thermal stability, high wear, and corrosion resistance. HEAs are first discovered in 1995 by Yeh et al. and coined as multicomponent alloys in 2004 by Cantor et al. [32–33]. Previous studies have shown that most of the HEAs

are composed of simple face-centered cubic (FCC), body-centered cubic (BCC), or FCC + BCC solid solutions owing to their high configurational entropy. The composition of these HEAs can be tailored to obtain promising properties such as high hardness and ductility as well as high resistance to wear, oxidation, and corrosion.

7.1 Thermodynamic calculations for fabrication of HEA

In accordance with the Hume-Rothery rules, the factors affecting the binary solid solutions, i.e., atomic size difference, valence electron concentration (VEC), the crystal structure of the solute and solvent atoms, and the difference in electronegativity, will be used to design the HEAs. The criteria adopted for the solid solution phase formation is given by various parameters according to the laws of thermodynamics. According to Zhang et al., the various parameters for HEA formation are given as follows [41, 42]:

$$\Delta S_{mix} = -R \sum_{i=1}^{n} C_i \, ln \, C_i \tag{3}$$

$$\Delta H_{mix} = 4 \sum_{i=1, i \neq j}^{n} C_i C_j \Delta H_{mix}^{AB} \tag{4}$$

$$\Omega = |T \Delta S_{mix} / \Delta H_{mix}| \tag{5}$$

$$r = \sum_{i=1}^{n} C_i r_i \tag{6}$$

$$\delta^2 = \sum_{i=1}^{n} C_i \left[1 - \frac{r_i}{\sum_{i=1}^{n} C_i r_i} \right]^2 \tag{7}$$

δ is the atomic size difference; C_i is the atomic fraction of ith element with radius r_i. T denotes the temperature. The average radius of all elements is r. The enthalpy change before and after mixing of A and B elements is ΔH_{mix}^{AB}, and ΔH_{mix} and ΔS_{mix} are the enthalpy change and mixing entropy of the HEA. The parameter Ω is the interaction parameter of the HEA. For HEA formation, $\delta \leq 6\%$ and $\Omega \geq 1.1$.

7.2 Applications of HEA

These novel HEAs find applications in various fields such as aerospace, automobiles, submarines, and nuclear and power plant industries [43–45]. Therefore, it is of utmost importance to develop a reliable and feasible technique of microjoining using these HEAs as fillers. The important conventional brazing methods cause cracking issues in joining metal-ceramic components due to the presence of various interfacial compounds, in addition to the inherent residual stress in fusion welding approaches. In contrast, brazing avoids the cracking issues and distortion, but still, the creation of numerous IMCs at the interface is a serious concern. HEAs with an optimal balance of strength and ductility and mainly solid solution phases may overcome these interfacial compounds and can be an excellent tool for brazing industries. There is not enough work done in this area related to high-entropy brazing. Few studies talk about the laser brazing of Ni superalloy. Gao and his coworkers used FeCoNiMnCu non-equiatomic HEA to braze the Ni superalloy [46]. In this study, they also evacuated the effect of brazing time and foil thickness on the shear strength and found a remarkable improvement.

7.3 Advantages of HEA as filler

There are various reasons for using HEA as fillers:

1. High ductility of HEAs will lead to a robust tough joint. It can be fabricated easily into wires and rods.

2. HEAs will minimize the formation of IMCs in the joint and ensure a safe joint.

3. The cracking and distortion are minimized due to the IMCs and reaction compounds compared to the fusion bonding processes.

4. A single solid solution of HEAs will have a narrow or even zero solidus-liquidus range which is beneficial for brazing to avoid distortion and thermal damage.

8. Future and current status of HEA fillers

These fillers are generally prepared by using various methods such as arc melting, powder metallurgy, sputtering, laser cladding, and electrodeposition. Most of the HEAs are produced initially by arc melting starting from Cantor alloy and its derivatives.

8.1 Arc melting

Arc melting consists of melting the pure elements by an arc in a vacuum or argon environment followed by a cooling approach [46]. However, some vaporizable elements are difficult to use in this method and lead to porosity. The schematic diagram is shown in **Figure 7**.

8.2 Bridgman solidification

There is another variation of the solidification method for obtaining single crystals of HEA, known as Bridgman solidification method [46]. In this case the polycrystalline HEA is melted and cooled from the liquid state (usually an eutectic
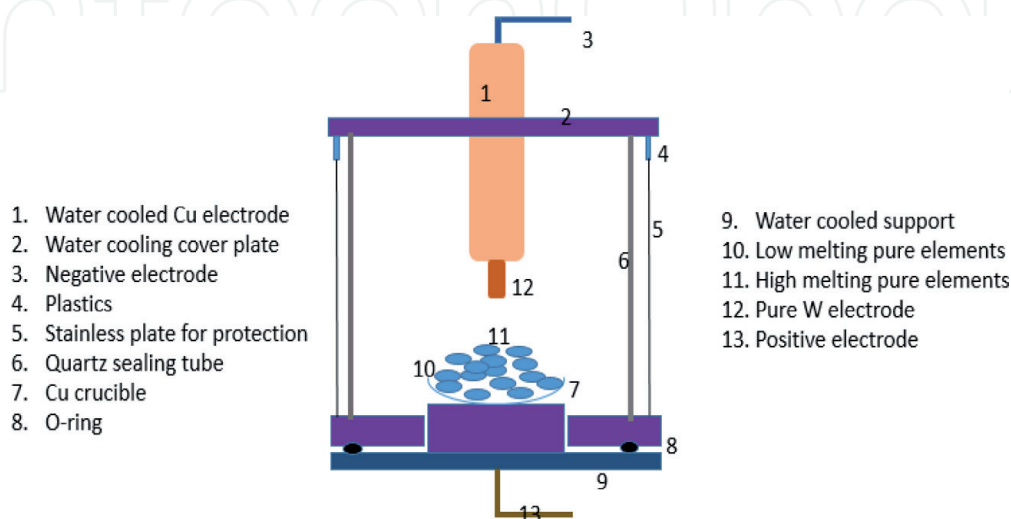

1. Water cooled Cu electrode
2. Water cooling cover plate
3. Negative electrode
4. Plastics
5. Stainless plate for protection
6. Quartz sealing tube
7. Cu crucible
8. O-ring
9. Water cooled support
10. Low melting pure elements
11. High melting pure elements
12. Pure W electrode
13. Positive electrode

Figure 7.
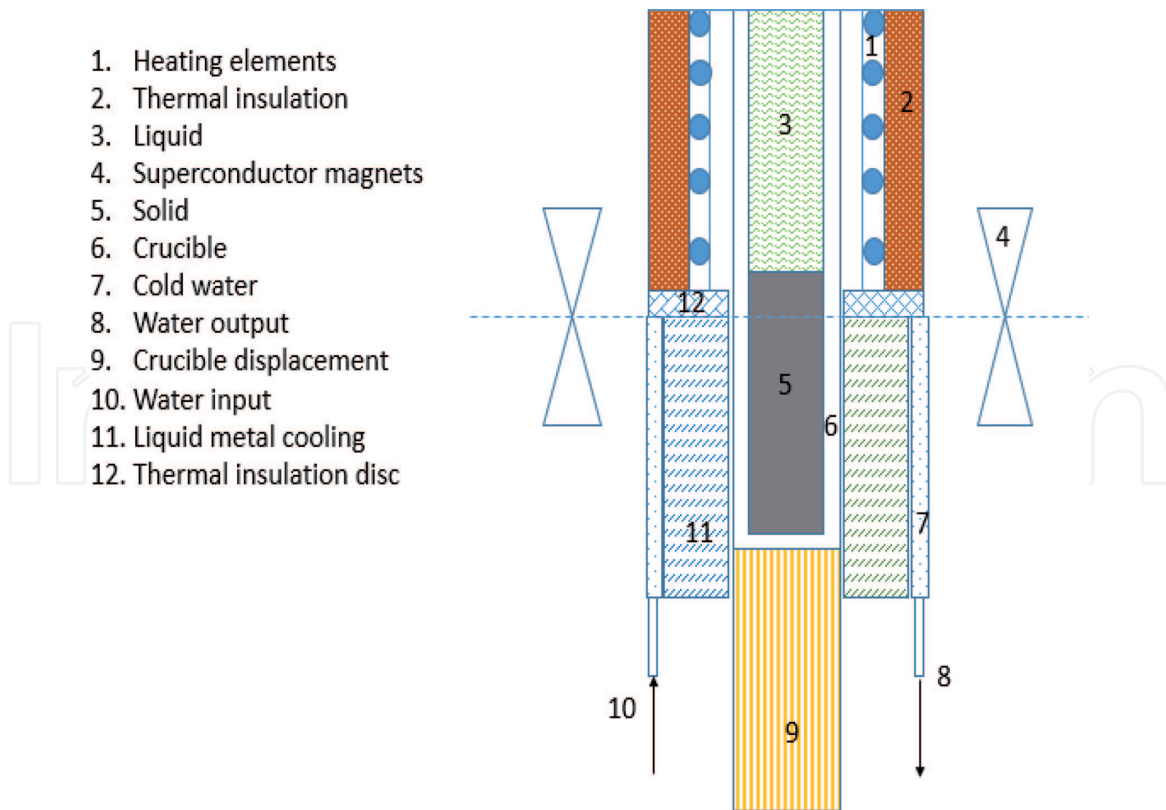*Schematic for arc melting.*

Figure 8.
Bridgman solidification.

Ga-In alloy) and stays in a liquid state at room temperature from the end of the container with a seed layer (**Figure 8**).

8.3 Powder metallurgy

Another important method is powder metallurgy, i.e., mechanical mixing of constituent powders followed by densification by heat treatment such as hot-pressing, cold isostatic pressing, or SPS treatment (**Figure 9**). The powder metallurgy method is beneficial in improving the homogeneity and distribution of powders.
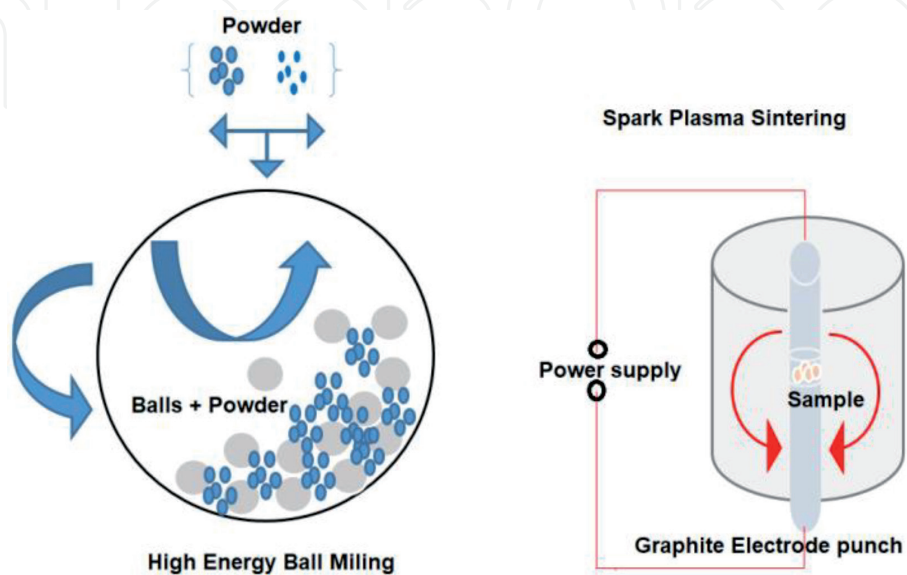

Figure 9.
Mechanical alloying and sintering diagram.

Heat treatment is used to prepare the bulk specimen which also relieves the stress in the powders to get the stable HEAs (**Figure 9**).

Varalakshmi and her co-workers produced nanocrystalline CuNiCoZnAlTi HEA with a BCC structure by powder metallurgy and showed a remarkable increment in the hardness and compressive strength of 8.79 GPa and 2.76 GPa, respectively, higher than the current hard-facing alloys [47]. Other researchers have also produced HEAs with sputtering, laser cladding, and electrodeposition in a few studies. However, until now, a wide exploration of these HEAs for microjoining is very limited.

9. Summary and guidelines

This HEA filler research has the ability to develop a new microjoining material which can join dissimilar materials (ceramic-metal, ceramic-superalloy, ceramic-steel, etc.) and will contribute to the integration of various metallic and ceramic components toward achieving high joint strength, protecting devices, and saving energy. This invention will largely improve the performance of the conventional IMC containing brazing fillers. The development of IMC free fillers for joining is the long-standing demand for microjoining industries. This high quality and the innovative project can be further expanded and commercialized to Korean companies looking for cheap and IMC-free Al alloys for joining for mass production. The combination of HEA filler with those of industry will provide the scale and credibility needed to bring high-entropy alloy technology fully into the market. Newly developed alloys will be patented and commercialized to global companies to realize a capital sum and raise the world economy.

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