

RECENT DEVELOPMENTS IN MECHANICAL ALLOYING

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Abstract. Mechanical alloying (MA) is a powder metallurgy processing technique that involves repeated cold welding, fracturing and rewelding of powder particles in a high-energy ball mill. Started in 1960 to produce oxide dispersion strengthened (ODS) nickel-based superalloys to obtain high strength both at room and elevated temperatures, this technique is now widely used to synthesize a variety of non-equilibrium alloys including solid solutions, intermetallics, and glassy alloys. Apart from the ODS alloys, the products of MA are now finding ever increasing and newer applications. The present article briefly summarizes the past achievements of MA followed by a discussion of the present activities. Three specific topics have been covered – nanocomposites, metallic glasses, and use of MA powder particles in metal combustion. The article concludes with an indication of the topics that need special attention in the near future.

1. INTRODUCTION

Mechanical Alloying (MA) is described as a high-energy milling process in which powder particles are subjected to repeated cold welding, fracturing, and rewelding. The transfer of mechanical energy to the powder particles results in introduction of strain into the powder through generation of dislocations and other defects which act as fast diffusion paths. Additionally, refinement of particle and grain sizes occurs, and consequently the diffusion distances are reduced. Further, a slight rise in powder temperature occurs during milling. All these effects lead to alloying of the blended elemental powders during the milling process. The result could be constitutional changes – formation of solid solutions (both equilibrium and supersaturated), intermetallic phases (equilibrium, metastable and quasicrystalline), and amorphous phases – or microstructural changes leading to development of ultrafine-grained and nanostructured phases. All these effects have been well documented in the literature and excellent reviews and monographs are available dealing with the different aspects [1,2].

As has been pointed out frequently, the technique of MA was developed out of an industrial necessity in 1966 to produce nickel-based superalloys that combined the room temperature strength obtained by precipitation hardening with the high-temperature strength achieved through dispersion hardening [3]. The historical development of MA during the last 40 years or so can be divided into three major periods. The first period, covering from 1966 to 1985, was mostly concerned with the development and production of oxide dispersion strengthened (ODS) superalloys for applications in the aerospace industry. Several alloys, with improved properties, based on Ni and Fe were developed that found useful applications. These included the MA754, MA760, MA956, MA957, MA6000, and others. Majority of this work was done at the INCO laboratories in the US. In addition to these, several small-scale applications were also developed for the MA products. A typical example is the Meals, Ready to Eat (MRE) used by the US military during the Gulf war and subsequently. The second period, covering from 1986 up to about

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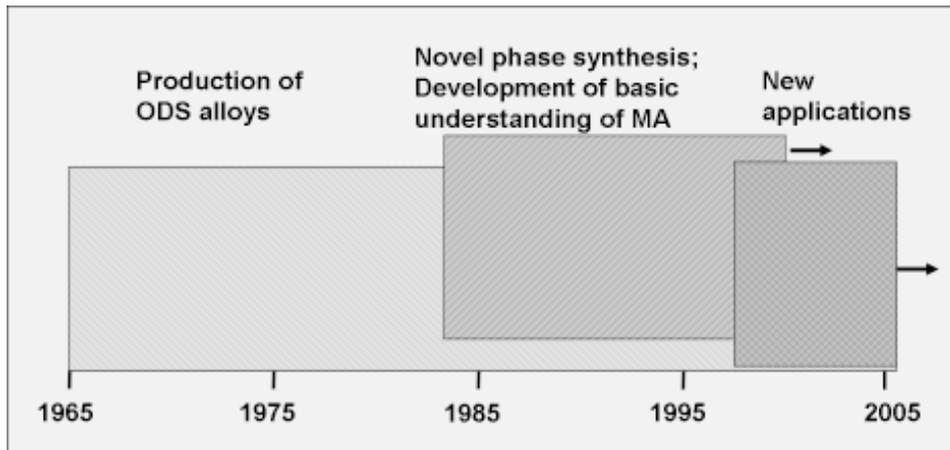


Fig. 1. Schematic showing the different periods of development in mechanical alloying (MA) since its inception in 1966.

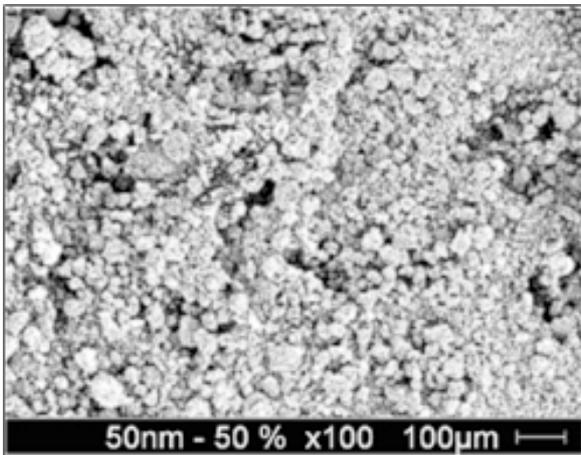


Fig. 2. SEM micrograph of Al-50 vol.% Al_2O_3 (50 nm) powder milled to the steady-state condition (20 h) showing a uniform distribution of Al_2O_3 in the Al matrix.

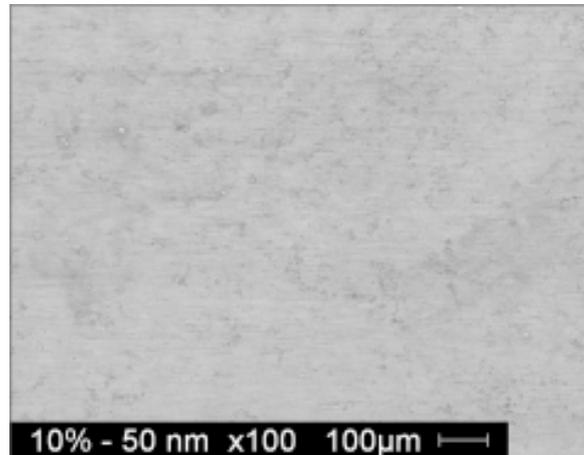


Fig. 3. SEM micrograph of Al-10 vol.% Al_2O_3 (50 nm) powder consolidated to close to 100% density by a combination of vacuum hot pressing and hot isostatic pressing.

2000, saw lot of developments in the fundamental understanding of the processes that take place during MA. Along with this, a large number of dedicated conferences were held and there was a burgeoning of publication activity [4]. Comparisons have been frequently made between MA and other non-equilibrium processing techniques, notably rapid solidification processing (RSP). It was shown that the metastable effects achieved by these two non-equilibrium processing techniques are similar. Revival of the mechanochemical processing (MCP) took place and a variety of novel substances were synthesized. Several modeling studies were also conducted to enable prediction of the phases produced or microstructures obtained, although with

limited success. The third period, starting from about 2001 saw a reemergence of the quest for new applications of the MA materials, with the realization that contamination of the milled powders is the limiting factor in the widespread applications of the MA materials. Innovative techniques to consolidate the MA powders to full density while retaining the metastable phases and/or nanostructures in them were also developed. All these are continuing with the ongoing investigations to enhance the scientific understanding of the MA process. Fig. 1 shows these periods of development schematically.

This paper attempts to summarize the present status of MA and put the broad field of MA in a

proper perspective regarding some of the recent and important developments in this field.

2. PAST DEVELOPMENTS

The technique of MA was developed in response to an industrial need to produce nickel-based superalloys that could be used at high temperatures in the turbine industry. After several trials, high-energy ball milling, now known as MA, was selected as the most appropriate processing method. Since then, a number of ODS superalloys have been developed through MA and these have found ready acceptance in the industry. At the moment, a large number of ODS alloys based on Fe and Ni are used in the industry and details of their compositions, properties, and methods of manufacture may be found in the literature [see, for example, refs. 1,2,5–7]. Apart from these major industrial applications, the MA products were also finding use, on a smaller scale, in meals, ready-to-eat (MRE) heaters, pigment and paint industry, as super-corroding alloys for releases in the ocean, for spray coating purposes, and to convert waste material into value added product.

The most important contribution of MA studies during the late 1980's and the 1990's was in trying to establish a firm scientific basis for understanding the process and the products that have formed by this technique. The generally accepted explanation for alloying to occur from blended elemental powders and formation of different types of phases is that a very fine and intimate mixture (often lamellar if the constituent elements are sufficiently ductile) of the components is formed after milling, if not the final product. The crystalline defects introduced into the material (grain boundaries, interfaces, dislocations, stacking faults, vacancies, and others) and a slight rise in the powder temperature were found to facilitate alloy formation. If the final desired phase had not formed by MA, then a short annealing treatment at an elevated temperature was found to promote diffusion and consequently alloy formation. Accordingly, it was shown that by a proper choice of the process parameters and alloy composition, it is possible to produce solid solutions (both equilibrium and supersaturated), intermediate phases (crystalline and quasicrystalline) and amorphous alloys. The thermodynamic criterion that the phase with the lowest free energy, under the continuous deformation conditions, would be the most stable phase has been found to be valid. A significant attribute of MA has been the ability to even alloy metals with

positive heats of mixing that are difficult to alloy otherwise.

Explanations were also provided for the formation of solid solutions in terms of the Hume-Rothery rules, so widely used to rationalize equilibrium substitutional solid solutions. These were found to be generally applicable [1,2,8,9], although not about the range of solubilities. Powders with fine grain sizes, and consequently a large grain boundary area, were shown to have a higher solid solubility level than the coarse-grained powders. Reasons for the formation of intermetallic phases (equilibrium and metastable crystalline as well as quasicrystalline) were also provided. An important contribution during this period was investigating reasons for the formation of amorphous phases in MA powders and also synthesis of bulk metallic glass compositions by MA [1,2]. The anticipation in this case was that if the glassy powders could be consolidated to full density without crystallization, then it would be possible to overcome the limitation of section thickness imposed by the solidification route. A number of investigations were also undertaken to develop aluminides based on Ti, Ni, and Fe to determine whether these alloys in the nanostructured condition would exhibit more ductility [10-15]. Many of these investigations were not encouraging since the MA-processed nanostructured aluminide alloys did not show any improved ductility at room temperature. However, among others, a composite consisting of 60 vol.% of Ti_5Si_3 and 40 vol.% of γ -TiAl alloy showed significant improvement in its deformation behavior and exhibited even superplastic behavior at 950 °C and at a strain rate of $4 \cdot 10^{-5} s^{-1}$ [16].

Significant amount of work was also devoted to modeling of the MA process with a view to predict phase formation [17]. Although the general trends of energy available in different types of mills [18,19] were predicted (the SPEX mills were the most energetic, the ball mills the least energetic, and the planetary mills in between), and some other trends, it was not possible to accurately predict phase formation. However, based on the available experimental results, the process could be modeled in the form of milling maps [19,20]. Thus, it appears that we have still a long way to go before we can reliably predict the nature of phases that form under the given conditions of milling.

3. CURRENT ACTIVITIES

As mentioned earlier, in recent years, there has been a resurgence of search for novel applications

Table 1. Mechanical properties of Al-Al₂O₃ nanocomposites obtained by milling and subsequent consolidation by vacuum hot pressing and hot isostatic pressing.

Al ₂ O ₃ Particle size	Volume fraction	Yield Strength (MPa)	Compressive Strength (MPa)	Elastic Modulus (GPa)	Elastic Modulus calculated by the rule of mixtures* (GPa)
50 nm	5%	488	605	78	83
50 nm	10%	515	628	90	95
150 nm	5%	409	544	75	83
150 nm	10%	461	600	77	95

* $E_c = V_m E_m + V_r E_r$, where E and V represent the elastic modulus and volume fraction, respectively and the subscripts c , m , and r represent the composite, matrix, and reinforcement, respectively.

of MA products. Apart from this, investigations are also continuing on developing newer and “better” materials through MA and also to understand the science behind the formation of these novel materials. As typical examples, we would like to discuss three specific MA investigations that are being actively pursued in our research group now. These include (1) development of homogeneously-dispersed nanocomposites, (2) formation of metallic glasses, and (3) use of metal particles in combustion.

3.1. Nanocomposites

Achievement of a uniform distribution of the reinforcement is essential in achieving good mechanical properties of composites. Further, the mechanical properties of the composite tend to improve with increasing volume fraction and decreasing particle size of the reinforcement. Traditionally, a reasonably large volume fraction of the reinforcement could be added, if the size of the reinforcement was large (on a micrometer scale). But, if the reinforcement size is very fine (of nanometer dimensions), then the volume fraction added was restricted to about 2 to 4%. However, if we are able to introduce a large volume fraction of nanometer-sized reinforcement, the mechanical properties of the composite are likely to be vastly improved. Therefore, attempts have been made to synthesize and characterize Al-Al₂O₃ composites with the Al₂O₃ reinforcement varying in size from 50 nm to 5 μm, and the volume fraction from 5 to 50%. MA of these powder blends for 20 h in a SPEX mill

was able to produce a very uniform dispersion of the fine 50-nm Al₂O₃ particles, up to a volume fraction of 50% (Fig. 2). Similar uniform distributions were obtained in the larger size and other volume fractions of the reinforcement also. The uniform distribution of the reinforcement phase was also confirmed through the X-ray elemental mapping technique [21].

These composite powders were very hard and strong and consequently it was not possible to consolidate them to full density by any of the presently available different techniques. Therefore, to determine the effect of reinforcement particle size and volume fraction, nanocomposites with 50 nm and 150 nm particle size and 5 and 10 vol.% were consolidated to full density. Even at these small volume fractions, full (close to 100%) density was achieved only by vacuum hot pressing followed by hot isostatic pressing (Fig. 3). Compression testing was done on the fully dense samples, and the strength properties and modulus values were determined. Table 1 lists the mechanical properties of these composites, from which it may be noted that the strength and modulus increased (a) with increasing volume fraction and (b) decreasing size of the reinforcement. Comparison of these modulus values with those calculated using the iso-strain and iso-stress conditions suggests that composites with the smaller reinforcement particle size behave closer to the iso-strain condition, while composites with the larger reinforcement size behave closer to the iso-stress condition [22]. Thus, it is possible to tailor the modulus of the composite by

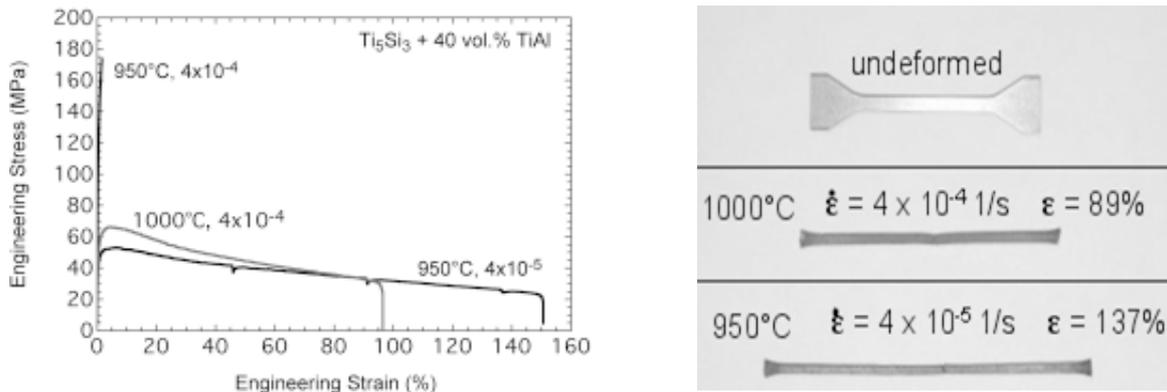


Fig. 4. (a) Stress-strain curve of γ -TiAl+60 vol.% Ti_5Si_3 composites showing that superplastic deformation was achieved at 950 °C and a strain rate of $4 \cdot 10^{-5} \text{ s}^{-1}$ and 1000 °C and a strain rate of $4 \cdot 10^{-4} \text{ s}^{-1}$. (b) Photographs of the actual tensile specimens before and after testing.

choosing the appropriate reinforcement size and volume fraction.

Composites of γ -TiAl and Ti_5Si_3 phase, with 60 vol.% of the silicide phase in it were produced by MA of the blended elemental powders. Fully dense and porosity-free compacts were produced by hot isostatic pressing, with a grain size of about 400 nm. Tensile testing of these composite specimens was conducted at different temperatures and strain rates. From the stress-strain plots of these composites (Fig. 4a), it is easy to note that these composites exhibit the phenomenon of superplasticity at temperatures as low as 950 °C and a strain rate of $4 \cdot 10^{-5} \text{ s}^{-1}$. Although the strain rate employed is relatively low, it is interesting that superplasticity was observed at 950 °C [23], corresponding to about $0.5 T_m$, where T_m is the melting point of the alloy; the coarse-grained material shows this behavior only at temperatures about 300-400 °C higher than the temperature observed in this investigation [24]. Fig. 4b shows the actual specimens before and after tensile testing.

3.2. Glass formation

Metallic glasses have been produced in several alloy systems by many different techniques. One of the most commonly used techniques is rapid solidification processing (RSP). As is well known, the critical cooling rate for glass formation needs to be exceeded for the glass to form. However, since this is an experimentally determined value

(and is also difficult to measure), several other criteria have been proposed for glass formation. These include (a) a high reduced glass transition temperature, $T_{rg} = T_g/T_l$, where T_g is the glass transition temperature and T_l is the liquidus temperature of the alloy, (b) presence of deep eutectics in the phase diagrams, (c) negative heat of mixing between the constituent elements, and for bulk metallic glass formation additionally (d) presence of at least three components, and (e) atomic radius difference of $>12\%$ between the elements, and some others [25]. We could also determine if other criteria could be formulated to predict glass formation in alloy systems. In this context, we had recently undertaken a comprehensive investigation on the glass formation behavior and stability of several Fe-based glassy alloys synthesized by MA.

A multicomponent $\text{Fe}_{60}\text{Co}_8\text{Zr}_{10}\text{Mo}_5\text{W}_2\text{B}_{15}$ alloy was milled from blended elemental powders and the X-ray diffraction patterns were recorded as a function of milling time. All the diffraction peaks expected of the constituent elements were seen at the start of milling. With continued milling, a glassy phase had formed, which on further milling started to crystallize, termed mechanical crystallization. Fig. 5a compares the XRD patterns at 20 and 50 h of milling and one can clearly see the relatively sharp (110) peak of Fe at 50 h of milling time. This is only the start of crystallization, but on external heat treating the glassy alloy powder for 1 h at 700 °C, almost complete crystallization occurred (Fig. 5b). This work clearly demonstrates that it is possible

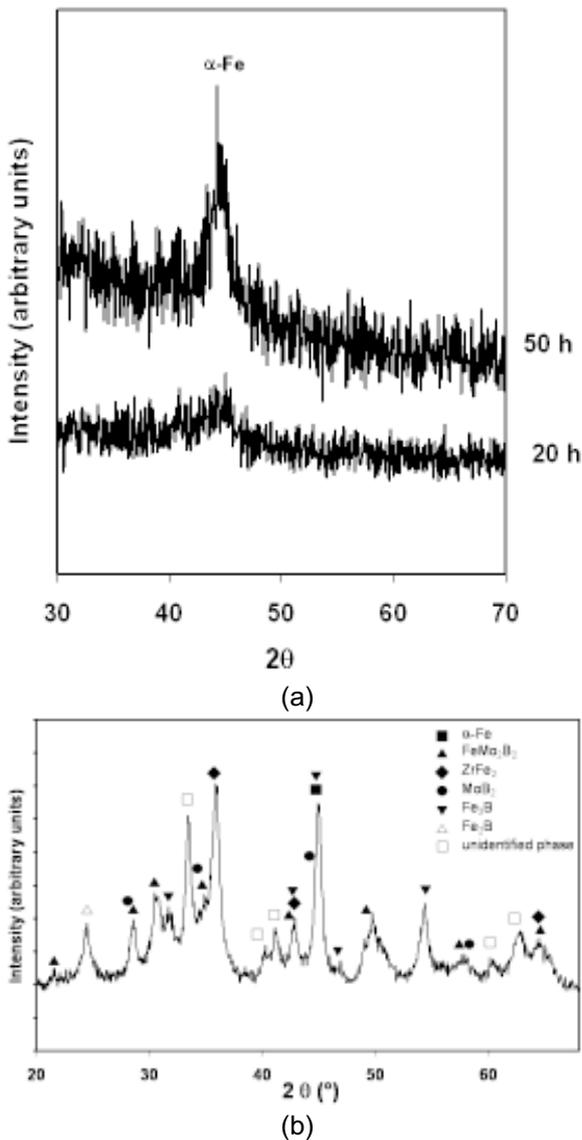


Fig. 5. (a) Comparison of the XRD patterns of $\text{Fe}_{60}\text{Co}_8\text{Zr}_{10}\text{Mo}_5\text{W}_2\text{B}_{15}$ blended elemental powders milled for 20 and 50 h, (b) XRD pattern of the amorphous powder annealed for 1 h at 700 °C showing almost complete crystallization.

to produce glassy phases of bulk glassy alloy compositions [26] and that by consolidating these powders without allowing crystallization to occur, one should be able to produce any size of the bulk glass product.

While searching for appropriate compositions to produce glassy phases, we had investigated several Fe-based alloys of the general composition $\text{Fe}_{42}\text{X}_{28}\text{Zr}_{10}\text{B}_{20}$ (where X = Al, Co, Ge, Mn, Ni, or Sn). It was noted that glass formation was easy

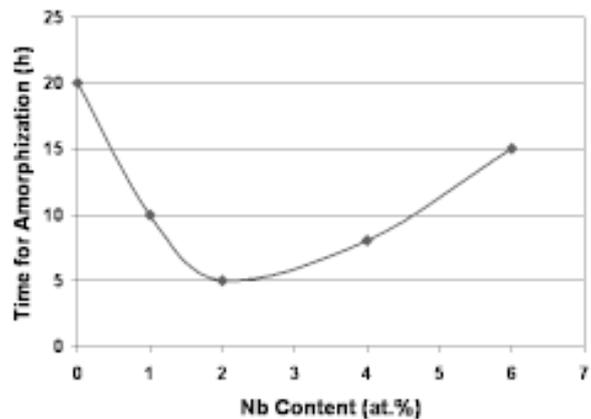


Fig. 6. Time required for formation of the glassy phase in mechanically alloyed $\text{Fe}_{42}\text{Ni}_{28}\text{Zr}_{10-x}\text{Nb}_x\text{B}_{20}$ alloys as a function of the Nb content. Note that the time required decreases with increasing Nb content up to certain level and then it increases again.

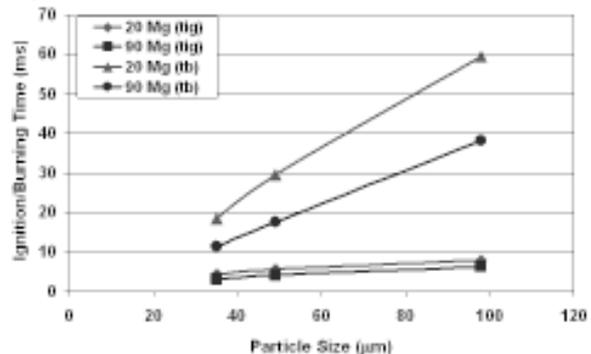


Fig. 7. Ignition time, τ_{ig} and burning time, τ_b as a function of particle size for two different alloy compositions.

(as determined by the short time required for glass formation) in alloys containing Al, Ni, or Ge, but not in other systems (Table 2). From an analysis of the phase diagrams of these alloy systems, it appears that the presence of a large number of intermetallic phases in the phase diagrams is conducive to formation of the glassy phase [27]. Again from the binary phase diagrams between these elements and Fe, it is apparent that those alloy systems that formed the glassy phase contain a number of intermetallic phases, whereas those that do not form the glassy phase easily have phase diagrams which feature a solid solution at room temperature. Thus, it is clear that the presence of a large number of intermetallic phases in the system helps in promoting the glassy phase formation.

Table 2. Time required for glass formation in $\text{Fe}_{42}\text{X}_{28}\text{Zr}_{10}\text{B}_{20}$ alloys and the number of intermetallics in the respective binary alloy systems

Element X	Milling time for glass formation (h)	Number of intermetallics between X and	
		Zr	Fe
Al	10	8	5
Co	No glass formation	5	None
Ge	10	5	5
Mn	No glass formation	1	None
Ni	20	7	1
Sn	No glass formation	3	2

It has also been recently reported [28,29] that addition of an alloying element that has a positive heat of mixing with one of the constituent elements promotes glass formation and also the plasticity of the glassy alloys. We had also noted that addition of Nb to an Fe-Ni-Zr-B glass-forming composition makes it easier to produce the glassy phase in Nb-containing alloys. Nb has a positive heat of mixing with Zr (+17 kJ/mol) and also that it has an atomic size significantly larger than Fe and Ni, but smaller than Zr. All these seem to increase the glass formability of the alloys [30]. However, this effect seems to be most prominent only at small concentrations of Nb; larger additions make the glass formation again more difficult (Fig. 6).

3.3. Combustion studies of metal particles

One of the potential applications for materials with fine grain sizes is in improving the combustion characteristics of propellants [31]. It is known that Mg can be easily combusted, but it has a low heat content (14.9 MJ/kg). On the other hand, Al has a higher heat content (32.9 MJ/Kg), but is more difficult to combust. Therefore, it was decided to see if Al and Mg can be alloyed together to increase the net heat content and also be able to combust relatively easily. Accordingly, a series of Al-Mg powder blends of different compositions were mechanically alloyed in a SPEX mill. Since the milled powder normally contains a range of particle sizes, the milled powder was sieved to well-defined particle sizes in the ranges of 32-38 μm , 44-53 μm , and 88-105 μm . These powder particles were then burned in a methane-air premixed flame on a Meeker burner, at a temperature of approximately

1,800K. Burning of the powder particles was recorded using chopped images by long-exposure digital photography. Knowing the rotational speed of the chopper and the number of exposures per revolution, the burning time, τ_b , and the ignition time, τ_{ig} , could be determined. Fig. 7 shows the plot of variation of τ_{ig} and τ_b with particle size for two different alloy compositions (Al-20 at.% Mg and Al-90 at.% Mg) [32]. It is clear from this plot that both the ignition and burning times decrease with (a) a decrease in particle size and (b) an increase in Mg content. Thus, by a judicious choice of particle size and alloy composition, it should be possible to control the ignition and burning times.

4. FUTURE OF MECHANICAL ALLOYING

From the above description of the type of activities, it becomes amply clear that the technique of MA is very efficient in synthesizing a variety of equilibrium and non-equilibrium materials. Some of the present issues that need immediate attention are that we need homogeneous, pure, macro-defect (i.e., porosity and cracks)-free and fully dense materials for obtaining reproducible results. Further, large-scale applications require materials that could be produced in tonnage quantities, and materials that have been well characterized and exhibit reproducible properties and also exploit them for commercial applications. But, the bane of MA has been the three C's – Cost, Consolidation, and Contamination. Powder processing is expensive and therefore, unless one is able to identify niche markets for the MA products and the powders produced in large volumes, cost is likely to continue to be high. However, in some cases, e.g., hydrogen

storage materials or magnetic alloys, the high cost may be acceptable in view of the improved properties and enhanced performance. Consolidation of the milled powder continues to be a serious problem. There have not been many investigations to report successful consolidation of MA powders to achieve full density and simultaneously retain the metastable effects. Newer and improved methods or modification/combination of the existing methods may be necessary [33,34]. The last point is that contamination of the MA powder has been a serious issue in many cases [2]. Some solutions have also been suggested to minimize/avoid contamination completely. These include enclosing the mills inside chambers that have been evacuated and/or filled with an inert gas, or using high-purity gaseous atmosphere in which the powders could be milled. Both options could be expensive and/or impractical.

The MA powders could also be used in the as-produced powder condition without consolidation. Examples for such applications include use as catalysts, pigments, solder, hydrogen storage materials, etc. In that case, some of the above problems may not be relevant. Further, even if a bulk component made of MA powder needs to be used, if that material could tolerate high gaseous impurities, then also there is no problem. But, with the intense research activity going on all over the world, there is great potential for MA products and bright future for research in this area.

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