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Synthesis of Titanium Oxide Particles Reinforced with Magnesium by Argon Controlled Stir Casting Process and Characterization

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Abstract The experimental investigations of magnesium based matrix reinforced with the TiO_2 particles with 2.5, 5, 7.5 and 10% weight ratios are presented in this research paper. The purpose of this work is to elaborate the manufacturing processes of pure magnesium metal with the addition of Titanium oxide through the vacuum stir casting route with the use of Argon as shielding gas to prevent the oxidation. The Electrical Discharge Machining (EDM) was used to resize the samples for conducting experiments. The mechanical parameters like density, hardness, tensile strength and ultimate tensile strength were evaluated by immersion test, Rockwell Tester and UTM respectively. The presence of particles in phase is ensured by XRD analysis. The microstructural examinations revealed that homogeneous distribution of reinforcing particles in magnesium matrix. The results also proved that improvement of 24% in Hardness, 29.8% in Ultimate tensile strength due to addition of particles.

Keywords Magnesium, Titanium Oxide, XRD Analysis, SEM Analysis, Mechanical Properties, Stir Casting

1. Introduction

The increasing challenges to achieve light weight of structural materials, magnesium alloys are potential candidates for automotive, aerospace applications. Magnesium castings have the potential to be used as replacements for many ferrous castings in power- train, chassis including parts of engines, brake, suspension and steering. The distinct advantages of magnesium over aluminium alloys are lower latent heat fusion that makes longer die life, faster solidification, higher machinability and more precise tolerances due to higher fluidity[1].The interest in magnesium alloys for automotive applications is based on the combination of high strength properties, low density durability, design flexibility and reducing relative investment[2]. The ability to produce the functional component at an affordable price is the real challenge for lightweight materials. The new materials are considered to be part of vehicle designs[1,3]. The high performance in demanding applications has led to research and development efforts in processing magnesium matrix composites with cost–effective fabrication technologies[4,5]. The excellent properties of magnesium promote its utilization in automobile-related products[6]. The defect free microstructures can be achieved by the use of stir casting process with more economic manner[7]. The Vacuum stir casting method prevented the entrapment of gases onto the melt and oxidation of magnesium during synthesis[8]. The max quantity of 30% particles added with magnesium and successfully fabricated by stir casting process [9]. The reinforcement particles should be stronger than matrix material to get desired properties[10].The preheating of reinforcement particles increase the surface energy and strength[11]. The clusters of particles, porosity and high oxidation were controlled by stirring temperature[12].The effects of adding yttrium with magnesium are investigated and found that the improvement in grain size, mechanical properties [13]. Argon is used for extinguishing fires in high-temperature industrial processes[14,15]. The Electrical discharge Machining is suitable for machining the composites without usage of tool [16]. Many researchers reported that reinforcements strengthen the matrix by imparting better mechanical and tribological properties[17,18]. The mechanical properties were improved by using stir casting process[19,20,21]. In this research the magnesium and titanium oxide particles were selected, because there were no systematic investigations and research about the addition of Titanium oxide or Titanium dioxide with magnesium.

2. Experiments

2.1. Sample Preparation

Magnesium is silvery-white alkaline earth metal and one third lighter than Aluminium. The properties of the matrix and reinforcement materials are listed in Table 1. Magnesium may be prepared from electrolysis of fused magnesium chloride, most repeatedly obtained from sea water. Pure titanium is a transition metal with a lustrous silver-white color and is resistant to corrosion including sea water and chlorine. Titanium has the highest strength to weight ratio of any metal. The vacuum stir casting method in stages is used to fabricate five samples. Table 2 represents the proportion of particles with matrix material. The experimental set up to produce the samples is presented in Figure 1.In the stir casting process the reinforcing phases usually in powder form are distributed into the molten magnesium by means of mechanical stirring. The raw materials used in experiments are represented in Figures 2 &3.The effect of high strength can be achieved by homogenous distribution of secondary particles in the matrix by stirring process. Otherwise uneven distribution can lead to premature failures in both reinforcement free and reinforcement rich areas. Since the magnesium alloy is highly sensitive to oxidation, there is a possibility of entrapment of gases and other inclusions in the stir casting process. This will further increase the viscosity of the molten metal and produce imperfections within the material. Thus the stirring process needs to be more astutely controlled for magnesium alloys than aluminum alloys in order to prevent the entrapment of unwanted gases and other inclusions. Since magnesium is a flammable material and easily gets oxidized in the presence of oxygen a shielding gas is required to control the atmosphere inside the furnace. The protection of this environment from the oxygen is prevented by the use of Argon. Argon is used for thermal insulation in energy efficient windows that the element undergoes almost no chemical reactions. The outer atomic shell makes argon stable and resistant to bonding with other elements. It is mostly used as an inert shielding gas in welding and other high-temperature industrial processes where ordinarily unreactive substances become reactive.

Table 1. Properties of matrix and reinforcement materials

Material	Magnesium	Titanium
Phase	Solid	Solid
Meltingpoint	650°C[923K]	1668°C[1941K]
BoilingPoint	1091°C[1363K]	3287°C[3560K]
Density	1.738g/cm^3	4.506g/cm^3
Heat of fusion	8.48kJ/mol	14.15kJ/mol
Heat of vaporization	128kJ/mol	425kJ/mol

Table 2. Percentage of particles in specimens

Sample	proportion
1	Pure magnesium
2	Mg+ 2.5 % TiO_2
3	Mg+ 5 % TiO_2
4	Mg+ 7.5 % TiO_2
5	Mg+ 10 % TiO_2

Figure 1. Experimental set up.

Figure 2. Magnesium raw material

Figure 3. Titanium oxide in weighing machine

The distribution of the particles in the molten matrix depends on the geometry of the mechanical stirrer, stirring parameters, placing of the mechanical stirrer in the melt, melting temperature, and the characteristics of the particles added. In our research work the matrix material is heated to above liquidus temperature and the allowed to cool in between the stage of semi-solid. The particles are preheated and then mixed with matrix material. Then the combinations of composites are heated again to above melting temperature of matrix material. The control panel set up to indicate the various temperatures to control the process is as shown in Figure 4. The porosity of melted metal matrix can be reduced by the use of Dichlorodifluoromethane with argon to create the protective atmosphere.

Figure 4. Control Panel setup

Figure 5. Prepared Samples

The produced final samples are shown in Figure 5. The first three samples represent the composites after machining and next two represent before machining stage.

2.2. Microstructure

2.2.1. XRD Analysis

The XRD patterns of the specimens prepared by stir casting process are as shown in Figures 6& 7. The samples are polished with mirror like surfaces with an automatic polisher. The phase analysis was carried out with a speed of 3 degree/ minute with a range of 0-100 degrees. As the intensities agree with the theoretical values, the increase in the peak areas gives the information about the kinetics of the reaction process. It means that the composite was formed with in the systems as per the XRD pattern shown in Figures 6& 7. The main diffraction peaks corresponding to the phases of Mg and Ti were detected. It is expected that the powder particle size can affect the process.it is observed that smaller particles of the elemental powder are more beneficial in the reaction between Mg and Ti. It is evident that $TiO_2$is formed completely and a large quantity of molten magnesium fully infiltrates through the aperture gap of the particulate.

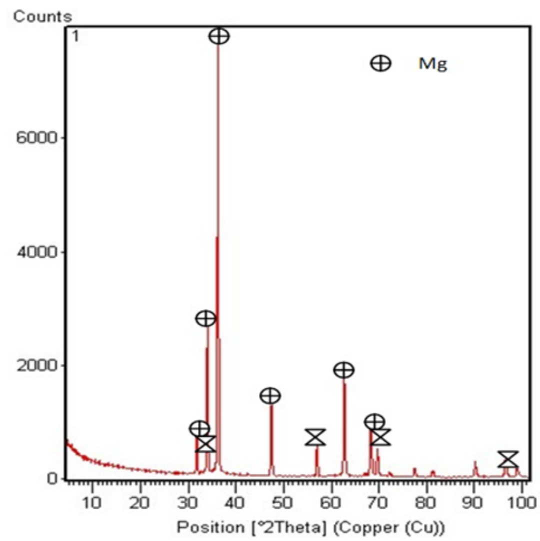

Figure 6. XRD pattern for sample 4

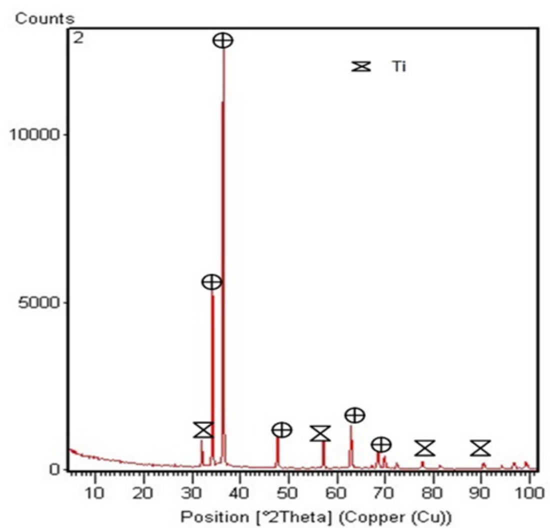

Figure 7. XRD pattern for sample 2

2.2.2. SEM Analysis

In order to characterize the microstructure of Mg-TiO_2 composites, Scanning Electron Microscope is used. The interface of Mg and TiO_2 is examined through the SEM. The grain size with its boundary of pure magnesium specimen is presented in Fig.8.

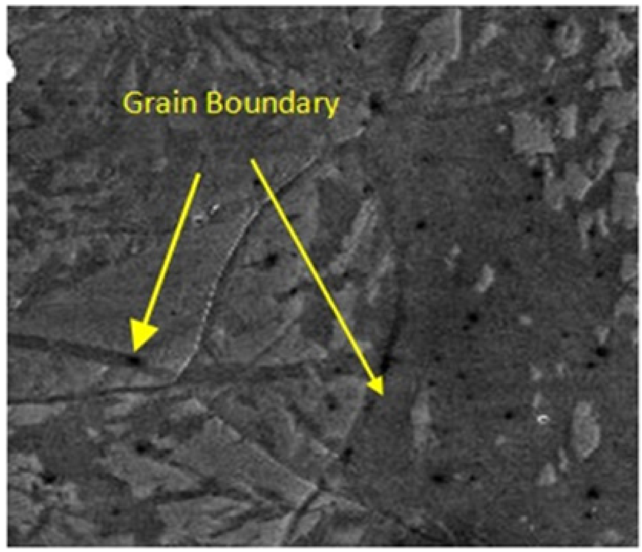

Figure 8. SEM Image for Pure Magnesium Sample

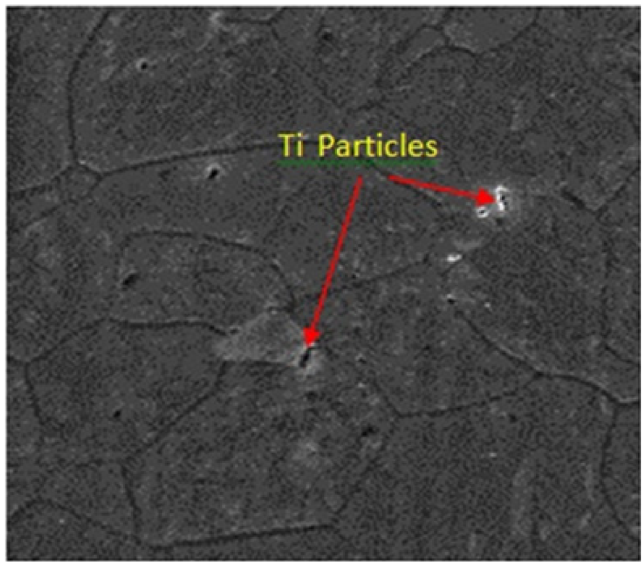

Figure 9. SEM Image for Mg+5% TiO_2 Sample

From the microscopic point of view the bonding within the magnesium matrix is ensured with less porosity. The image reveals the presence of small amount of TiO_2particles which is shown in Figure 9. It is noted that free interference of the components are strongly connected and precipitate obtained. The particle of TiO_2 in variation in sizes that is not soluble in magnesium matrix is presented in Figure 10.

2.3. Mechanical Testing

The Density of extruded specimens was estimated with

Archimedean principle, by determining the specimen mass and volume, and basing on the apparent loss of weight after immersing the specimen in water. The sample is as shown in Figure 11. Hardness tests of the fabricated composite materials were made by using Rock well harness tester. Five indentations were made on the transverse section diameter for specimens produced by the stir casting process. Then the average values of each specimen are taken into account. Static compression and tensile tests of the fabricated composite materials were made with the ASTM Standard B557-06 is followed at room temperature. The examined test pieces in the tensile have an overall length of 65 mm and gauge length 15 mm. The prepared cylindrical tensile specimens are as shown in Figure 12. The Yield stresses (YS) and ultimate tensile strength (UTS) was determined employing at least two specimens for each combination by using standard Universal Testing Machine.

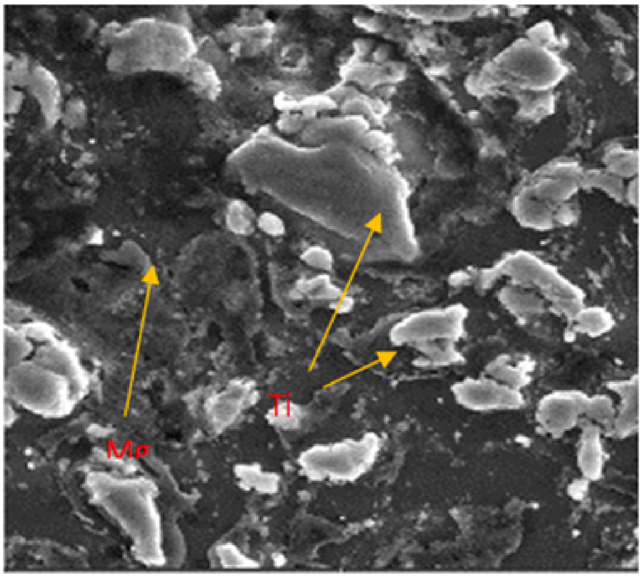

Figure 10. SEM Image for Ti particles in Mg Matrix

Figure 11. Sample in density Test

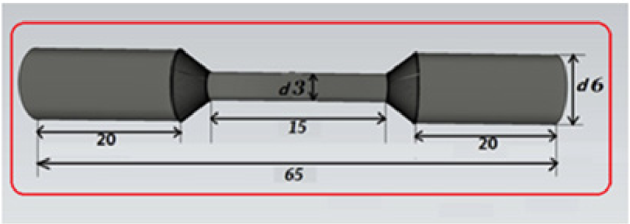

Figure 12. The ASTM standard tensile test specimen

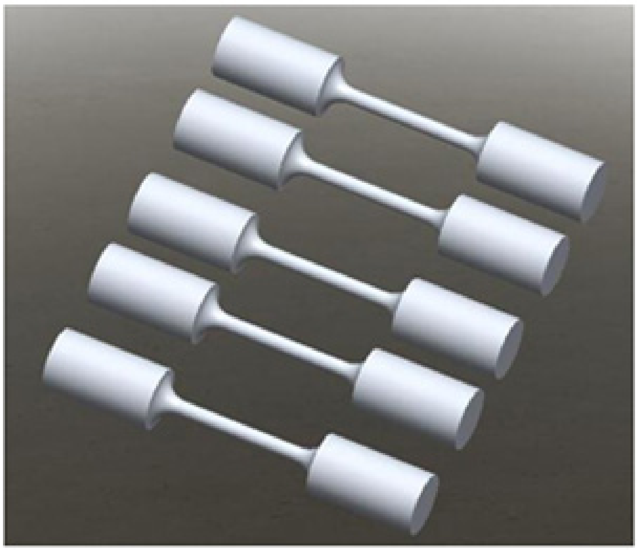

Figure 13. Tensile test samples

3. Results and Discussions

The actual density measurements of each samples and their comparison with theoretical values are shown in Figure 14. The differences between the real and theoretical densities indicate the presence of porosity. Hardness tests of the fabricated composite materials revealed its diversification depending on the weight ratios of the reinforcing particles in the Magnesium matrix. The values of investigated composite materials are characterized by the higher hardness compared to the non-reinforced material. Hardness of composite materials increases with increasing content of the reinforcing material in the metal matrix is presented in Figure 15.The reduced stiffness and strength of the magnesium alloys set a limit on its applications in the field of automobile and aerospace industries. Magnesium alloy composites can overcome such difficulties with improvement in mechanical properties were proved by addition of hard particles in matrix alloys. The values of Ultimate tensile strength and yield strength for the fabricated samples by vacuum stir casting are listed in the 16. It reveals that there were significant improvements in UTS and YS due to grain refinement of particles and matrix. It ensured that there was perfect interfacial bonding between the matrix and reinforcements.

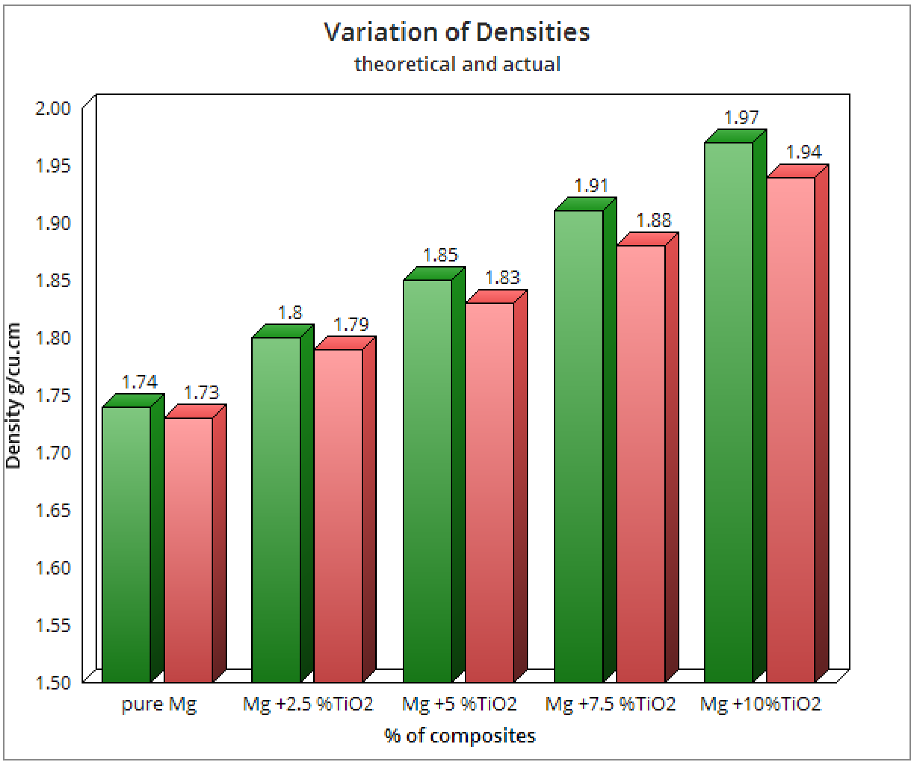

Figure 14. Porosity results

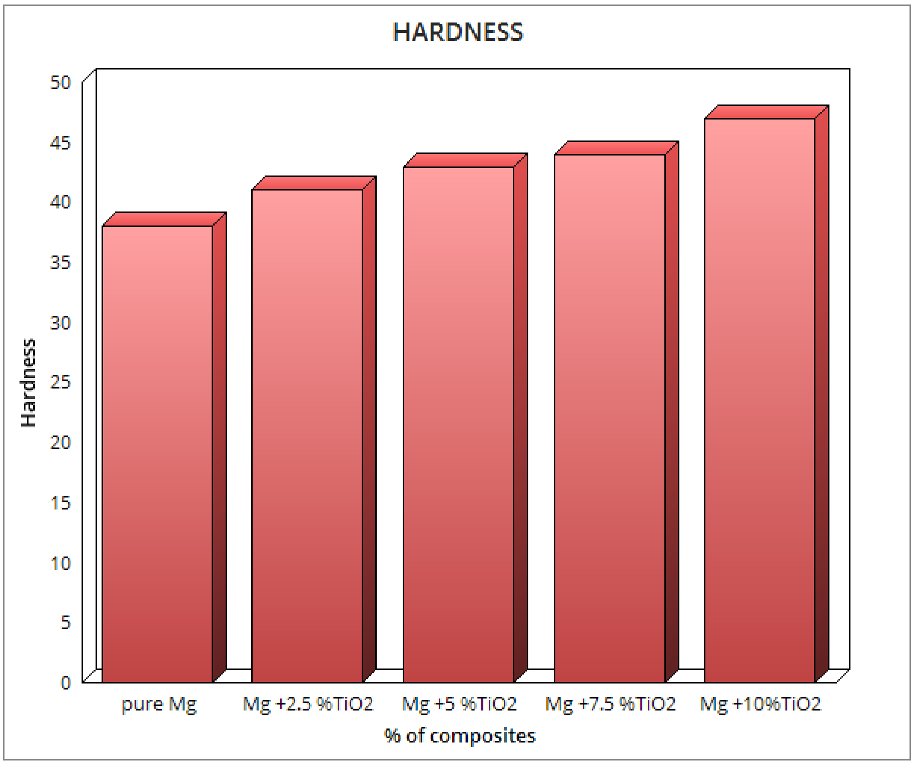

Figure 15. Hardness values

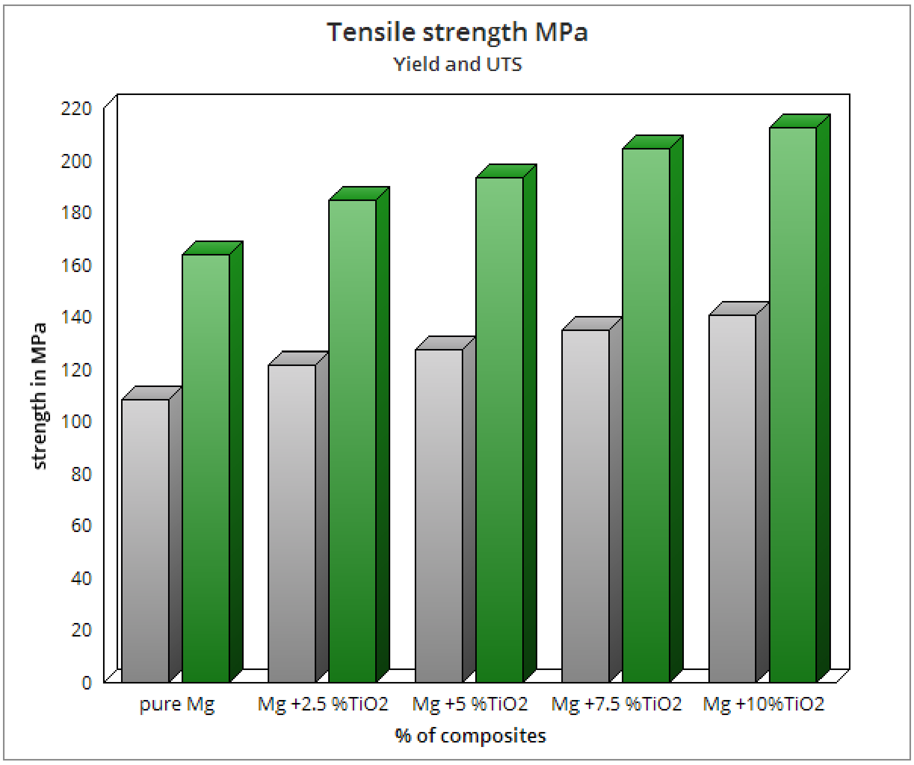

Figure 16. Tensile strength

From the experimental investigations it is concluded that as follows.

- The Vacuum stir casting method is one of the cost effective methods with protected argon atmosphere and easy process to disperse the TiO_2 in the Magnesium matrix.
- Due to the presence of TiO_2, the morphology of the Mg phase is changed to discontinuous and fine. There are no imperfections in the interfacial bonding between the matrix and particles.
- The uniform distribution of particles into the matrix is ensured by the investigations of SEM.
- The values of density for the prepared composite materials are near to the theoretical one but existing differences indicate presence of porosity.
- The porosity test revealed that stir casting method in stages with argon atmosphere is suitable for preparing Mg-Ti composites with very less porosity.
- The improvement of mechanical properties of composites is attributed to the grain refinement of matrix as well as particles.
- The Hardness, Yield strength and Ultimate Tensile Strength of composites were increased to significant level due to addition of reinforcement particles.
- The addition of the TiO_2 particles of the reinforcing material to the magnesium matrix increased the expected hardness of the composite materials and got the value of 17.8% more than the unreinforced material.

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