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## Effect of Al<sub>2</sub>O<sub>3</sub> Nanoparticles as Reinforcement on the Wear Properties of A356/Al<sub>2</sub>O<sub>3</sub> Nanocomposites Produced by Powder Metallurgy

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# Effect of Al<sub>2</sub>O<sub>3</sub> nanoparticles as reinforcement on the wear properties of A356/Al<sub>2</sub>O<sub>3</sub> nanocomposites produced by powder metallurgy

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## ABSTRACT

*In this study, microstructure and wear properties of A356 aluminium matrix nanocomposites reinforced with nano-Al<sub>2</sub>O<sub>3</sub> particles were investigated. Powder metallurgy method was used for the production of 1 wt.% and 2 wt.% nano-Al<sub>2</sub>O<sub>3</sub> particle reinforced nanocomposites. After 1 hour of mechanical milling of A356 and nano-Al<sub>2</sub>O<sub>3</sub> powders, green compacts were obtained by cold pressing. Green compacts were sintered at 550°C and vacuum environment (10<sup>-6</sup> mbar) for 1 hour. Samples were characterized by density, hardness measurements, scanning electron microscopy investigations and wear tests. As the reinforcement ratio increased, there was a decrease in the densities of the nanocomposites, as well as an increase in the porosity. The highest hardness and the lowest weight loss values were obtained in 1wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites. A decrease in hardness was measured at 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites.*

## INTRODUCTION

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32  
33 Aluminium matrix composites (AMCs) are produced by adding particles with  
34 different properties into the Al alloy by various methods in order to be used in  
35 applications requiring functional properties. In particle-reinforced aluminium  
36 composites, Al alloy is generally defined as the matrix and additional particles are  
37 defined as the reinforcement phase. It is possible to produce composites with different  
38 properties by reflecting the specific properties of the matrix and reinforcement  
39 materials to the composite material. AMCs reinforced with SiC, Al<sub>2</sub>O<sub>3</sub> (alumina), B<sub>4</sub>C,  
40 TiB<sub>2</sub>, ZrO<sub>2</sub>, SiO<sub>2</sub> and graphite particles are frequently used in automotive and aerospace  
41 applications due to their superior mechanical and physical properties [1]. AMCs are also  
42 reinforced with other synthetic, recycling and natural reinforcement materials such as  
43 tetragonal zirconia polycrystalline [2], Si<sub>3</sub>N<sub>4</sub> [3], fly ash, rice husk ash [4-6]. With the  
44 powder metallurgy method, particle reinforced AMCs are produced at lower  
45 temperatures than liquid production methods (such as infiltration and Stir casting). In  
46 addition, as the reinforcement particles can be distributed more homogeneously in the  
47 matrix, the desired properties are obtained more efficiently [7]. Carbide, boride and  
48 oxides are often used as reinforcement materials in particle reinforced composites [8-  
49 12]. The improvement in the mechanical properties of AMCs is directly related to the  
50 type, size and reinforcement ratio of the reinforcement particles. [13]. When the  
51 reinforcement particles used in the production of AMCs are nano-sized, the ductility of  
52 the composite is preserved as well as the improvements in strength and hardness [14].  
53 Kang et al. [15] states that Al<sub>2</sub>O<sub>3</sub> reinforcement increases hardness and mechanical  
54 properties in Al/ Al<sub>2</sub>O<sub>3</sub> (between 1-7 vol. %) nanocomposites produced by powder

55 metallurgy method. Rahimian et al. [16] reported that the hardness of Al/  
56 Al<sub>2</sub>O<sub>3</sub> composites produced by powder metallurgy method increased, and the best yield  
57 strength was obtained from composites with a reinforcement ratio of 10 wt.%. Thereby,  
58 in many studies, properties such as optimum hardness, mechanical strength, ductility,  
59 microstructure morphology, depending on the amount of reinforcement phase or  
60 process parameters were investigated in nano AMCs produced by powder metallurgy  
61 [7,17]. Ezatpour et al. [13] produced A7075/ Al<sub>2</sub>O<sub>3</sub> nanocomposites with 0-1.2 wt.%  
62 reinforcement ratios by stir casting method and then applied extrusion process and  
63 investigated microstructure and mechanical properties. They reported that the  
64 extrusion process with nano-particle reinforcement increased the mechanical  
65 properties. Manivannan et al. [18] reported that A6061/1,2 wt.% SiC nanocomposites  
66 produced by ultrasonic cavitation assisted stir casting method were harder and showed  
67 better wear resistance than A6061 alloy. In addition, the amount of reinforcement  
68 phase in nano aluminium composites is an effective parameter on hardness and relative  
69 density [19]. Tiku et al. [20] produced Al/ Al<sub>2</sub>O<sub>3</sub> (1-4 wt.%) composites with Al<sub>2</sub>O<sub>3</sub>  
70 particles synthesized by sol-gel technique. They reported that as the reinforcement ratio  
71 increased, the hardness increased, but the abrasion resistance decreased. Soltani et al.  
72 [21] manufactured AMC by addition of 10Ce-TZP (tetragonal zirconia polycrystal) / Al<sub>2</sub>O<sub>3</sub>  
73 nanoparticles (1-10 wt.%) via powder metallurgy. The results showed that the wear  
74 resistance increased up to 7 wt.% reinforced samples and decreased with further  
75 reinforcements. Deaquino-Lara et al. [22] studied on the tribological characterization of  
76 Al7075/graphite composites produced by mechanical alloying and hot extrusion. They

77 produced their samples using different reinforcement ratios (0- 1,5 wt. %) and  
78 mechanical milling times (0-10 hours). They concluded that hardness and wear  
79 resistance improved by adding 1,5 wt.% graphite and mechanical milling of 10 hours.

80 It has been stated in many studies that the reinforcement phase in particle  
81 reinforced composites improves the mechanical properties of the matrix alloy [7,15,18].  
82 There is a limited number of studies examining microstructure and wear performance of  
83 nano-  $\text{Al}_2\text{O}_3$  added A356 alloys used in automotive and aviation industry. On the other  
84 hand, it is revealed that increasing the amount of reinforcement phase, increases the  
85 mechanical properties and wear performance of the composites, but after a certain  
86 amount, these properties tend to decrease with effects such as agglomeration [19]. It  
87 has also been reported that wear properties are adversely affected by nano-particle  
88 reinforcement and the wear rate increases with increasing reinforcement amount [20].  
89 Yildirim et al. are expressed that the most obvious difference between conventional  
90 particle-reinforced composites and nano-AMCs is the decrease of some mechanical  
91 properties such as hardness and wear behavior in case of increase over a critical ratio in  
92 nano-AMCs [23]. It is clearly seen in the literature that nanoparticle reinforcement has  
93 variable effects on wear properties. For this reason, there is a need for more work in  
94 this area. In this study, nano-  $\text{Al}_2\text{O}_3$  reinforced A356 aluminium matrix nanocomposites  
95 (AMnCs) were produced by powder metallurgy. The microstructure and hardness of the  
96 AMnCs were investigated. In addition, the wear performance of nanocomposites was  
97 investigated in a pin-on-disc type wear device. The microstructures of the composites  
98 and the worn surfaces were investigated by SEM/EDS.

99 **MATERIALS AND METHOD**

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A356 aluminum alloy powders used as matrix material in this study were obtained from LPW technology Ltd. (particle size range; 20-63  $\mu\text{m}$ ), nano-  $\text{Al}_2\text{O}_3$  powders used as reinforcement phase (purity: >99%, average particle size; 20 nm) were obtained from Nanografi Co. Ltd. A356 and nano-  $\text{Al}_2\text{O}_3$  powders were prepared by weighing them with a precision balance (precision; 1:10000 g). A356 alloy, 1 wt. % and 2 wt. % nano-  $\text{Al}_2\text{O}_3$  reinforced nanocomposites were milled for 1 hour in a single chamber Fritsch planetary ball mill with a capacity of 225 ml. Stainless steel balls with a diameter of 8 mm were used in the mechanical milling (MM) process. The ball/powder ratio was determined as 1:10 and the chamber was filled 50% and MM was carried out at a milling speed of 400 rpm. Prior to MM, 1% stearic acid was added to the chamber as a process control chemical. In order to prevent overheating, the milling process was applied with a 15-minute interrupt for every 15 minutes. Nano-  $\text{Al}_2\text{O}_3$  (reinforcement) powders were milled in ethanol for 5 minutes before mechanical milling of the nanocomposites because it is difficult to disperse the nanoparticles using the conventional grinding process. After MM process, AMnC powders were cold pressed (520 MPa) to obtain cylindrical green compacts with a height of 8 mm and a diameter of 12 mm. Green compacts were sintered at 550  $^{\circ}\text{C}$  in vacuum environment ( $10^{-6}$  mbar) for 1 hour. Heating and cooling rates were kept constant at 4  $^{\circ}\text{C}$  per minute. Density measurements of sintered AMnCs were made according to Archimets' principle using a PRECISA XB200h density measurement kit. The densities of 3 different samples of each reinforcement ratio were measured and averaged. Hardness measurements were performed on a

122 SHIMADZU micro-hardness instrument (200gf – 15 sec). Mean values at each  
123 reinforcement ratio were calculated by averaging three different measurements  
124 performed on three samples. Carl Zeiss Ultra Plus Gemini scanning electron microscope  
125 (SEM+EDS) instruments were used in AMnCs' characterization studies. Wear tests were  
126 performed on a standard pin-on-disc wear device with AISI 4140 (mean 64 HRC) steel  
127 disc. Tests were performed at room temperature and in dry conditions in accordance  
128 with ASTM: G99 [24] standards. A356/ Al<sub>2</sub>O<sub>3</sub> nanocomposites were tested with 30N  
129 load, 1ms<sup>-1</sup> sliding speed and 4 different sliding distances (500, 1000, 1500 and 2000 m).  
130 Each test was repeated three times and weight loss values were measured before and  
131 after the test. Sample surfaces were cleaned with alcohol before measurements. The  
132 samples were weighed on a precision scale with a resolution of 10<sup>-4</sup>g before and after  
133 wear tests, to determine weight losses. Wear rates were calculated using following  
134 equation.

$$W_a = \frac{\Delta G}{D.P.S} \quad (1)$$

136  
137 In equation (1), W<sub>a</sub> is the wear rate (mm<sup>3</sup>/Nm); ΔG is the weight loss (g); P is the  
138 load (N); S is the sliding distance (m) and D is the density (g/cm<sup>3</sup>). SEM analyses were  
139 carried out to examine the effects of wear on the worn surfaces.

## 140 **RESULTS AND DISCUSSIONS**

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### 142 **Density and Porosity**

143

144 The relative densities of the nanocomposites are shown in Figure 1. It was observed that  
145 with the increase in the amount of reinforcement, there was a decrease in the densities  
146 of AMnCs and an increase in the porosity values. Considering these results given in  
147 Figure 1, it is understood that the amount of reinforcement has a significant effect on  
148 the density. This is likely due to the application of MM. MM starts with cold welding of  
149 powder particles that are squeezed between two balls and/or between a ball and the  
150 container wall, as high-energy balls crash into one another and the container wall during  
151 the process. Then, powder particles undergo work hardening and in the last phase,  
152 particles are fragmented. Previous studies reported a slight increase in the powder  
153 dimension with the effect of cold-welding taking place in the first stage of the  
154 mechanical alloying process [25]. While the size of the powder particles is reduced by  
155 deformation with MM, the hardness of the powders increases with the work hardening.  
156 In a previous study, it was reported that the compressibility of powders decreased with  
157 the MM process [26]. In this case, the presence and increase in the amount of hard  
158 reinforcement particles leads further deformation by locating among the ball-powder-  
159 ball and ball-powder- container wall in the milling chamber. Thus, the hardness of the  
160 powders that undergo plastic deformation with MM increases further. Increasing the  
161 hardness of the powders reduces the compressibility by locking each other during  
162 pressing. Therefore, it can be said that the amount of reinforcement has an increasing  
163 effect on the porosity. In Al/ nano-  $\text{Al}_2\text{O}_3$  composites produced by powder metallurgy  
164 method, due to the high hardness of nano-  $\text{Al}_2\text{O}_3$  particles, it reduces the relative



165 density of the composites compared to the alloy by reducing the compression capacity  
166 [16].

### 167 **Microstructure**

168  
169 SEM images of the A356 alloy and AMnCs are given in Figure 2. The microstructure of  
170 the A356 alloy is given in Figure 2.a. In Figure 2.b, the structure was filtered and grain  
171 boundaries, pores and secondary phase particles were emphasized. Figure 2.c and  
172 Figure 2.d show the microstructures of 1 wt.% Al<sub>2</sub>O<sub>3</sub> and 2wt.% Al<sub>2</sub>O<sub>3</sub> nanocomposite  
173 materials, respectively. Secondary phase particles were observed in Figure 2.b (blue)  
174 and in all microstructures (light gray). The EDS results indicate that these secondary  
175 phase particles contain a high amount of silicon. Therefore, these phases are considered  
176 to be the silicon phase. In addition, the relatively high Al and O amounts in the EDS data  
177 for Figure 2.c (Point 5) and Figure 2.d (Point 4) indicate that these points may be regions  
178 with Al<sub>2</sub>O<sub>3</sub> reinforcements. Besides, Figure 3 shows the nano-sized alumina particles  
179 were distributed in the microstructure. EDS analysis which was applied at 80.000x  
180 magnification level reveal that point 1 and point 2 contains considerable amount of Al  
181 and O.

### 182 **Hardness**

183  
184 The hardness graph of AMnCs' is given in Figure 4. While the average hardness of the  
185 A356 alloy samples was 42.4 HV, the average hardness of 1 wt.% and 2 wt.% Al<sub>2</sub>O<sub>3</sub>  
186 reinforced nanocomposites were measured as 61.4 HV and 54 HV, respectively.  
187 According to the hardness results, the lowest hardness was obtained in the matrix  
188 material, while the highest hardness was obtained in 1 wt.% nano- Al<sub>2</sub>O<sub>3</sub> reinforced

189 composite. While the hardness of 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced composites is higher than that  
190 of A356 alloy, it is lower than 1 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced composites. This increase in  
191 hardness of 1 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites can be explained by the Orowan  
192 mechanism [13]. Reinforcement particles inhibit dislocation movement, causing an  
193 increase in dislocation density, thus increasing the stresses required for plastic  
194 deformation. Yildirim et al. [23] state that there are four reasons for this behavior of  
195 nano-AMCs; particle dimensions, particle shape, inter-surfaces formed between  
196 matrix/nano-particle, and agglomeration. The tendency to decrease in the hardness of 2  
197 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced composites is also thought to be due to their lower relative  
198 density compared to other composites. In a previous study, it was reported that  
199 increasing the reinforcement ratio from 1-3 vol% to 5% vol% in A6061- alumina  
200 nanocomposites, the hardness decreased depending on the decrease in the relative  
201 density [19].

## 202 **Wear properties**

203

204 It has been observed that the weight loss values increase almost linearly with the  
205 increase of the sliding distance in A356 matrix materials and AMnCs. In Figure 5, the  
206 weight loss results of A356 alloy and A356/ Al<sub>2</sub>O<sub>3</sub> nanocomposites were given. It is  
207 understood that the weight losses of A356/ Al<sub>2</sub>O<sub>3</sub> nanocomposites are lower than the  
208 matrix material A356 alloy. These results show that the wear performance of AMnCs is  
209 better than the wear performance of the A356 alloy. It was observed that the  
210 reinforcement ratio had a varying effect on the average weight loss. It has been  
211 determined that there is an average weight loss of 11.7 mg in the A356 matrix alloy.

212 With 1 wt.% reinforcement, a significant reduction in average weight loss was obtained  
213 (7.625 g). However, when the reinforcement ratio was 2 %, this value (11.4) increased  
214 again. It is clearly seen that 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites are subject to  
215 higher weight loss than 1 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites. This may be due to the  
216 differences in density and hardness values. AMnC with 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforcement  
217 showed higher hardness compared 1 wt.%. In addition, it is understood that the weight  
218 loss results of the 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites and the hardness results  
219 given in Figure 4 support each other. Significant fluctuations in wear rates are often due  
220 to variations in hardness [19]. Relatively higher porosity amounts in 2 wt.% Al<sub>2</sub>O<sub>3</sub>  
221 reinforced composites may adversely affect wear performance. During the wear test,  
222 fractures may occur at the edges of the pores. Therefore, weight loss tends to increase.  
223 It was also reported that the decrease in the relative density amount in A6061/ alumina  
224 composites causes a significant decrease in hardness, which is the main reason for the  
225 upward trend in wear rate [19]. In addition, wear debris is known to play an abrasive  
226 role and increase wear [20]. It has been stated that wear resistance is related to  
227 hardness, but the relationship between wear rate and microstructure of the material is  
228 not considered [27]. It is also reported that the wear resistance of AMCs reinforced  
229 above 7wt.% by weight decreases and this is due to increased porosity, particle  
230 aggregation, interfacial properties and thermal expansion difference [21].  
231  
232 Figure 6 shows the friction coefficient results of aluminium alloy and nanocomposites.  
233 When the weight loss and wear rate results given in Figure 6 are correlated with the

234 friction coefficient results, it is understood that 1 wt.%  $\text{Al}_2\text{O}_3$  reinforced nano  
235 composites, which have the lowest weight loss and wear rate, have the lowest friction  
236 coefficient. The force generated by friction is highly dependent on the hardness of the  
237 specimen. The higher hardness of the nano-composite sample compared to the matrix  
238 alloy A356 sample causes smaller plastic deformation on the nano-composite sample  
239 surface and reduces the friction coefficient [18]. The 1 wt.%  $\text{Al}_2\text{O}_3$  reinforced samples  
240 showed better wear performance as they were harder than the 2 wt.%  $\text{Al}_2\text{O}_3$  samples. In  
241 other words, 1wt.%  $\text{Al}_2\text{O}_3$  reinforced composites have lower friction coefficient values  
242 than 2 wt.%  $\text{Al}_2\text{O}_3$  reinforced composites. This is due to the low relative densities of 2  
243 wt.%  $\text{Al}_2\text{O}_3$  reinforced nanocomposites. Particles detached from the porous regions  
244 during wear cannot go out of the tribological system and increase the friction  
245 coefficient. In AMCs, hard reinforcement particles expand on the material and act as a  
246 lubricating layer against abrasives. By preventing direct contact of metallic surfaces,  
247 both the coefficient of friction and wear rate are reduced [21]. In Figure 7, SEM images  
248 of worn surfaces of alloy and  $\text{Al}_2\text{O}_3$  reinforced nanocomposites after 2000 m sliding  
249 distance are given. When the SEM images of the worn surfaces are examined, areas of  
250 ruptures, micro-cracks, delaminations, grooves and cavities can be seen during the wear  
251 test.

252

253 Worn surfaces of A356 alloy (Figure 7.a-c), A356/1 wt.% (Figure 7.d-f) and A356/2 wt.%  
254  $\text{Al}_2\text{O}_3$  (Figure 7.g-i) reinforced nanocomposites are shown in Figure 7 detailly. It is known  
255 that plastic deformation that occurs with cavities and grooves on the material surface is

256 a characteristic of abrasive wear [15]. The wear with wide ruptures and cracks are  
257 thought to be the results of adhesive wear mechanism. It can be seen that ruptures and  
258 cavities are more dense especially in the A356 matrix shown in Figure 7.a, b and c. This  
259 is obviously due to the fact that the hardness of the A356 matrix alloy is lower than that  
260 of the composites. The low hardness of A356 alloys could lead adhesive wear  
261 mechanism and the ruptures and cavities were generated by adhesion. It is seen that  
262 the grooves formed parallel to the sliding directions are less on worn surfaces of 1 wt.%  
263 Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites. Abrasive wear mechanism clearly shown in Figure  
264 7.d-f with infrequent cavities, ruptures and delaminations for 1 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced  
265 nanocomposites. It is observed that material loss and delamination due to abrasion  
266 increase in 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites. Micro-pores in the material act as  
267 a trigger for the formation and growth of micro-cracks on the wear surface. Micro-  
268 cracks grow with advancing sliding distances, causing partial ruptures from the material  
269 surface. The higher porosity value of 2 wt.% Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites also  
270 explains the high material loss. It has also been stated in some previous studies that  
271 plastic deformation and delamination occur with abrasive wear in SiC and alumina  
272 particle reinforced composites [28,29]. Porosity, agglomeration and inter-facial  
273 properties could be the reasons why wear resistance decrease for AMCs with higher  
274 reinforcement ratios than a critical ratio. That critical ratio depends on the size, type and  
275 the distribution of the reinforcement in the matrix.

## 276 **CONCLUSIONS**

277

278 The wear performance of AMnCs produced by reinforcing nano-  $\text{Al}_2\text{O}_3$  into A356  
279 aluminium alloy were investigated and the general results obtained from the study are  
280 given below.

- 281 • A decrease in the relative densities of AMnCs was observed with the increase of  
282 the reinforcement ratio, while an increase in the porosity values was observed.
- 283 • In SEM microstructures and EDS examinations, it was determined that there  
284 were second phase particles containing high amount of silicon in the structure.
- 285 • While the highest hardness was obtained in 1wt.%  $\text{Al}_2\text{O}_3$  reinforced  
286 nanocomposites, it was determined that there was a decrease in hardness when the  
287 reinforcement ratio was increased to 2 wt.%.
- 288 • The wear loss values increased proportionally with the sliding distance. In wear  
289 tests, the weight loss of A356/  $\text{Al}_2\text{O}_3$  composites was lower than that of the matrix  
290 material, A356 alloy. The lowest weight loss was obtained from 1wt.%  $\text{Al}_2\text{O}_3$  reinforced  
291 nanocomposites.

292

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294

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300

#### 301 **NOMENCLATURE**

302	$W_a$	wear rate ( $\text{mm}^3/\text{Nm}$ )
303	$\Delta G$	weight loss (g)
304	P	load (N)
305	S	sliding distance (m)
306	D	density ( $\text{g}/\text{cm}^3$ )

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### Figure Captions List

- Fig. 1 Mean relative density values of A356/alumina nanocomposites
- Fig. 2 SEM images of microstructures of A356/ alumina nanocomposites; A356 alloy (a), A356 color filtered (b), A356/1 % alumina composite (c) and A356/2 % alumina composite (d)
- Fig. 3 SEM image and EDS analysis of the microstructures of nanocomposites
- Fig. 4 Hardness of A356 alloy and alumina reinforced nanocomposites
- Fig. 5 Weight loss (a) and wear rate (b) of aluminium nanocomposites tested at different sliding distances
- Fig. 6 Friction coefficients of A356 alloy and alumina reinforced nanocomposites depending on the sliding distance.
- Fig. 7 SEM images of worn surfaces of A356 alloy and Al<sub>2</sub>O<sub>3</sub> reinforced nanocomposites after 2000 m sliding distance a,b,c) A356 alloy, d,e,f) A356/1 wt. % alumina, g,h,i) A356/2 wt. % alumina

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