EFFECT OF INGOT SIZE ON MICROSTRUCTURE AND PROPERTIES OF THE NEW ADVANCED AD730TM SUPERALLOY

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Abstract

The enhancement of efficiency in gas turbine engines requires the development of new superalloys capable of withstanding higher temperatures. The development of new industrial cast and wrought (C&W) disk alloys with required combination of strength, creep and fatigue properties at 700 °C is highly desired due to expensive cost of powder metallurgy. The development of U720Li in the nineties provided an intermediate solution in term of cost / properties combination between 718 and powder metallurgy superalloys. However, U720Li is known to be difficult to be manufactured due to its high γ' content (45%) and can be considered as the limit between C&W route and powder metallurgy route. Because of this and its intrinsic raw material content, U720Li is significantly more expensive than alloy IN718. To remain competitive on worldwide aeronautical market, engine designers are more and more challenged to optimize cost/properties combination of each component. In this context, Aubert&Duval has developed a new C&W superalloy, AD730TM, which was designed to offer a better combination between high temperature properties at 700 °C and cost compared to U720Li. The lower content of Ti and the lower γ' amount in AD730TM compared to U720Li is expected to reduce the segregation tendency. Contrary to U720Li, the better ability of AD730TM for C&W route enables the vacuum arc remelting of larger ingots up to 635 mm diameter, which is expected to have a beneficial impact on billet cost. This paper describes the work performed within the European Cleansky project CESAME (Cost Effective Superalloy for Advanced Modern Engine) which evaluated the feasibility of larger AD730TM ingots (635 mm diameter ingots) in order to improve AD730TM manufacturing cost. Comparisons between AD730TM ingot of 635 mm diameter (called in the following text AD730TM large ingot), AD730TM ingot of 508 mm diameter (called in the following text AD730TM small ingot) and U720Li ingot of 508 mm diameter (called in the following text U720Li ingot) were performed after conversion to billets of 254 mm but also on close-die forged disks. Results on billets showed that increasing ingot size for AD730TM alloy had a beneficial impact on billet microstructure and ultrasonic testing results in comparison with AD730TM and U720Li smaller ingots (508 mm diameter). Increasing AD730TM ingot diameter up to 635 mm was not harmful on chemical homogeneity but led as expected to an increase of carbonitrides size. Tensile, creep and fatigue tests on close-die forged disks showed no detrimental influence of AD730TM larger ingot size (635 mm diameter) compared to AD730TM smaller ingot (508 mm diameter). However, homogeneous microstructure and better UT detectability than those on U720Li disks were obtained on AD730TM disks. Mechanical properties of AD730TM and U720Li are very similar with advantages on tensile strength at high temperature and creep

elongation for AD730TM. Finally, fatigue tests on close-die forged disks showed that the largest carbonitrides observed on disk from AD730TM large ingot did not lead to detrimental effect on fatigue properties. This work was a reliable demonstration of AD730TM larger ingot feasibility to enhance the combination between cost and properties.

Introduction

The latest design of high-efficiency engines has high requirements for mechanical properties and temperature capability of the key components, especially the turbine disks. Alloy development for turbine disks with higher properties and temperature capability is consequently crucial in order to improve the thermal efficiency in gas turbine engines. In addition to higher properties, manufacturing cost optimization is also one of the major criterion to select a grade for engine designers. Powder metallurgy superalloys were developed for turbine disk application to withstand a higher temperature (N18 [1], Rene88DT [2], RR1000 [3]). However, the powder metallurgy process route is significantly more expensive than the conventional cast and wrought (C&W) route. Consequently, powder metallurgy superalloys remain an expensive solution for disks at highest temperatures like HPT applications with a minimum of required cooling air. Development and progress were made, especially with the development of the VIM-ESR-VAR triple melt route, which enabled the manufacture of higher gamma prime fraction superalloys by the conventional C&W route. In this context the alloy U720Li was developed. It is one of the highest alloyed disk materials that can be processed with the conventional C&W route. This alloy exhibits an improvement in temperature capability compared to previous superalloys such as IN718 and Waspaloy [4]. Nevertheless, U720Li remains a high expensive superalloy because of its well-known industrial issues. The high contents of Al and Ti elements in U720Li lead to a heavy precipitation of γ' phase with the presence of large γ/γ' eutectic precipitates in the as-cast microstructure which are difficult to dissolve after the homogenization heat-treatment [4]. It is one of the reasons why maximum VAR ingot diameter of U720Li is commonly limited to 508 mm. Workability issues of U720Li [4-5] require expensive forging processes with exclusively subsolvus deformation. The new C&W disk alloy AD730TM developed by Aubert&Duval was designed to offer a better combination of high temperature properties at 700 °C and cost compared to U720Li [6]. AD730TM chemistry was designed to decrease the intrinsic raw material cost regards to U720Li and to be more suitable to scraps recycling. Contrary to U720Li, AD730TM contains Nb and Fe (Table 1) which allow recycling, for example, of IN718 or other superalloys [7]. The lower γ' amount in AD730TM, which improves the ability for the C&W route, should also enable the melting of larger

ingots. The present work was performed within the European Cleansky project CESAME (Cost Effective Superalloy for Advanced Modern Engine), to evaluate the feasibility of larger AD730TM ingots (635 mm diameter ingots) in order to improve the AD730TM manufacturing cost. In this work, special attention was paid on properties potentially impacted by increasing ingot size such as noise level during ultrasonic inspection, macro and micro-segregation, fraction and size of carbides and nitrides, and low cycle fatigue properties. Tensile tests at elevated temperatures and creep tests were performed to precisely evaluate the effect of ingot size and fully compare AD730TM with U720Li. This paper describes the results obtained with this study.

	Ni	Fe	Co	Cr	Mo	W	Al	Ti	Nb	B	C	Zr
AD730 TM Base	4	8.50	16.0	3	2.7	2.3	3.5	1.1	0.02	0.02	0.03	
U720Li Base	-	15	16	3	1.25	2.5	5	-	0.02	0.02	0.03	

Table 1: AD730TM and U720Li typical chemistry compositions

Experimental Procedure

Nickel base alloys AD730TM and U720Li were used in this study (Table 1). Two AD730TM ingots with diameters equal to 508 mm and 635 mm were remelted from the same VIM heat (Vacuum Induction Melting). In the following text, these ingots will be respectively named AD730TM small ingot and AD730TM large ingot. Both ingots were triple melt by Electro-Slag Remelting (ESR) and Vacuum Arc Remelting (VAR). AD730TM ingots were then converted to 254 mm diameter billets. Same conversion steps were used for both ingots. Consequently, the amount of drawing deformation was more important for the billet forged from AD730TM large ingot (635 mm diameter) than that of the billet forged from AD730TM small ingot (508 mm diameter). U720Li billet of 254 mm diameter manufactured from triple melted 508 mm diameter ingot was supplied by Allvac company (ATI 720TM) and was used for alloy comparison. AD730TM billets were then forged to full scale low pressure turbine (LPT) disks (Figure 1) with an external diameter close to 500 mm and equivalent diameter close to 50 mm. These disks were close-die forged below γ' solvus temperature in the 1050 °C - 1090 °C temperature range. As reference, the same LPT disks were also forged from U720Li billet. AD730TM disks were solution heat-treated at 1080 °C for 4 hours and then oil quenched before being aged at 730 °C for 8 hours and air cooled. U720Li disk was solution heat-treated at 1100 °C for 4 hours then oil quenched before being aged as follows 650 °C / 24h + 760 °C / 16h.

The vacuum arc remelting (VAR) process of AD730TM was computed for each size of ingot by using the simulation software 'SOLAR' (which stands for SOLidification during Arc Remelting). This numerical model of the VAR process, using a finite volume method, has been developed for more than 20 years at the Nancy School of Mines (Jean Lamour Institute) [8, 9], in close collaboration with industrial partners (TIMET, CEZUS, AUBERT&DUVAL), to simulate remelting of titanium and zirconium alloys, nickel-based superalloys and high quality special steels. This 2D-axisymmetric model solves the coupled transient heat, momentum and solute transport equations, assuming a turbulent regime of the liquid metal flow in the melt pool of the remelted ingot. It is able to predict the temperature, composition and liquid pool profiles in the ingot at any time and takes into account electromagnetic stirring, buoyancy flows and turbulence effects. The mathematical model has already been

validated by comparison of calculated and experimental liquid pool profiles for nickel based superalloys and special steels ingots produced by Aubert & Duval [10, 11]. Ultrasonic Testing (UT) inspection was performed on both AD730TM billets with the same requirements. Top and bottom slices were cut to observe the microstructure and to perform mechanical tests. Carbides distribution was established by image analysis on Olympus microscope (BX61) device and with AnalySIS software on all billets. Micro-segregation was precisely assessed by microprobe analysis (CAMECA SX100) on all billets. Around 374 chemical analyses were performed following a line type "crenel" which the length was 10mm and the width of the crenel 1.7 mm. A tension of 15kV with a current of 20 nA and a LPET crystal was used for titanium analyses. The cooling rate of disks during oil quenching after solution heat-treatment was calculated in the 1050 °C - 700 °C temperature range within the disk by a Forge2® modeling and was estimated to be close to 220 °C/min (Figure 2). Gamma prime precipitates were observed after mechanical polishing to a mirror surface finish and selective etching using a field emission gun SEM (Supra 55).

Figure 1: Photography of LPT disk close-die forged

Figure 2: Cooling rate after solution heat-treatment calculated in the temperature range of 1050 °C - 700 °C within the disk by a Forge2® modeling – Position n°1 equal to 290 °C/min and Position n°2 equal to 174 °C/min

Results on billets

The vacuum arc remelting process was modelled to evaluate the effect of ingot size and melting parameters on the solidification. Figure 3 allows the comparison of the LST (Local Solidification Time) patterns for AD730™ small ingot and AD730™ large ingot. The results indicate that the LST is raised by 85% for AD730™ large ingot: from 30 minutes (AD730™ small ingot) to 55 minutes (AD730™ large ingot). The LST reaches its maximum value at the center location and decreases toward the periphery (Figure 4). As expected, the LST keeps growing with the ingot diameter: the surface to volume ratio gets smaller and the cooling rate is reduced. Therefore, trend to coarser structure, banding, large carbide size and freckle defect formation is more predominant when the ingot size is increased. Consequently, to evaluate the feasibility of AD730™ large ingot, special attention was paid to chemical homogeneity within the 635 mm diameter ingot in order to make sure that segregation tendency was acceptable. The influence of larger ingot size on carbonitrides size was also carefully evaluated. Increasing the local solidification time on 635 mm diameter ingot is expected to increase the size of carbonitrides especially in the core of the billet (Figures 3 and 4) and may have an impact on the fatigue properties. Finally, microstructure and ultrasonic testing results were compared on AD730™ materials to evaluate the influence of larger ingot size.

Figure 3: Modeling with Solar software of local solidification time for two size of AD730™ ingot: 508 mm (AD730™ small ingot) and 635 mm (AD730™ large ingot) diameters

Figure 4: Modeling of local solidification time vs. Ingot radius for AD730™ small ingot (508 mm diameter) and AD730™ large ingot (635 mm diameter)

Chemical homogeneity

As shown in Table 2, chemical analyses were performed on top and bottom positions of each billet. Good chemical homogeneity is observed for both AD730™ billets and no influence of larger ingot size was noticed. Additional assessment of chemical homogeneity was made by solution heat-treatment performed at temperatures close to the γ' solvus to highlight micro-segregation on both AD730™ ingots. Same results were obtained for both ingots (Figure 5). Duplex microstructures and homogeneous coarse grains microstructure were respectively observed with solution heat-treatments at 1100 °C and 1110 °C (Figure 5). The γ' solvus temperature was also measured by dilatometry on a Netzsch (402C) device. Samples (25 mm in length and 3.7 mm in diameter) were heated up to 1160 °C and then cooled down to room temperature with a heating and cooling rate of 3 °C/min. For each sample, two successive cycles were made. Derivative curves for heating and cooling obtained on the second cycle are given on Figures 6 and 7. These tests show that γ' solvus temperatures are close to 1127 °C for both AD730™ ingots and that γ' precipitation during cooling occurs below 1082 °C. Differences between γ' solvus temperatures determined by dilatometry tests and by heat-treatment are known to be due to different heating rates which were used for the tests. All these tests clearly indicate that the chemical homogeneity of both ingots is comparable.

		Ni	Fe	Co	Cr	Mo	W	Al	Ti	Nb	B	C	Zr
AD730™ small ingot	Top	Base	4.1	8.51	15.7	3.04	2.73	2.31	3.39	1.13	0.01	0.011	0.033
	Bottom	Base	4.1	8.50	15.7	3.04	2.72	2.3	3.41	1.13	0.01	0.011	0.033
AD730™ large ingot	Top	Base	4.1	8.49	15.6	3.04	2.72	2.27	3.41	1.13	0.01	0.01	0.03
	Bottom	Base	4.1	8.50	15.6	3.03	2.74	2.29	3.38	1.12	0.01	0.01	0.034
U720Li small ingot	Top	Base	-	14.5	15.9	2.98	1.25	2.52	5.02	-	0.01	0.01	0.033
	Bottom	Base	-	14.5	15.9	2.98	1.25	2.54	5.08	-	0.02	0.01	0.042

Table 2: Chemical composition measured on billets for the three materials

Figure 5: Microstructure obtained after heat-treatments close to γ' solvus - AD730TM small ingot: a) 1100 °C / 4h / AC – b) 1110 °C / 4h / AC – AD730TM large ingot: c) 1100 °C / 4h / AC – d) 1110 °C / 4h / AC

Figure 6: Dilatometry test to determine γ' solvus temperatures during heating

Figure 7: Dilatometry test to determine temperature range of γ' precipitation during cooling

Micro-segregation was also precisely assessed by microprobe analysis for the three materials. The segregation assessment was based on titanium which is the most segregated element. Results were analyzed to fit a reverse normal law and a segregation ratio, which is the ratio of standard deviation to the average value, was also calculated for each material (Figure 8). Micro-segregation of billet forged from AD730TM large ingot was slightly higher than that of the billet forged from AD730TM small ingot but similar to that measured on U720Li billet.

Figure 8: Cumulative fractions and segregation ratio of Ti element measured by microprobe analysis for the three materials

Carbonitrides

Carbides analyses were performed at the rim, mid-radius and center of top and bottom slices from billets. Results obtained at the center of each billet are shown in Figure 9. Largest carbonitrides were found on billet forged from AD730TM large ingot. The effect of ingot position on carbonitrides size was evaluated with AD730TM large ingot (Figure 10). The largest carbonitrides were measured in the top, then in the middle and finally in the bottom of the billet which is consistent with the modelling of the local solidification time (Figure 3). The local solidification time in the ingot bottom is shorter than in the top due to the high cooling effect of the VAR baseplate.

Figure 9: Carbonitrides analysis performed in the center of slices from the middle of the billets for the three materials

Figure 10: Carbonitrides analysis performed on billet from AD730™ large ingot in three positions (Top, Middle, and Bottom) in the center

Sonic inspection/Microstructure

Similar microstructures were observed on AD730™ and U720Li billets with an average grain size of 11 μm and the presence of unrecrystallized grains (Figure 11). Nevertheless, the amount of unrecrystallized grains observed on billet from AD730™ large ingot was much lower than those observed on billet from AD730™ small ingot (Figure 11). The higher amount of drawing deformation used for the conversion of 635 mm diameter ingot to 254 mm diameter billet might have reduced this amount of unrecrystallized grains. This lower amount of unrecrystallized grains has also promoted a lower noise level measured by ultrasonic testing on billet.

Figure 11: Microstructure observed at mid-radius of AD730™ and U720Li: billets forged from: a) AD730™ small ingot, b) AD730™ large' ingots and c) U720Li small ingot

Summary of billets results

This study on billets showed that increasing ingot size for AD730™ alloy had a beneficial impact on billet microstructure and ultrasonic testing results in comparison with AD730™ small ingot and U720Li. Increasing AD730™ ingot diameter up to 635 mm was not harmful on chemical homogeneity but leads as expected to an increase of carbonitrides size. The effect of AD730™ ingot diameter and carbonitrides size on fatigue

properties was evaluated on close-die forged disks to demonstrate the feasibility of AD730™ larger ingot.

Results on LPT disks

The second step of materials comparison was achieved on LPT disks which were close-die forged from the different billets (AD730™ small ingot, AD730™ large ingot and U720Li) and heat-treated.

Microstructure/gamma prime precipitation

Microstructure mappings of disk were performed on each material. Typical microstructure of U720Li is a banding microstructure with areas of fine grains ($\sim 40 \mu\text{m}$) and coarser grains ($\sim 110 \mu\text{m}$) (Figure 12c). AD730™ microstructures for both disks are, on the contrary, more homogeneous than those of U720Li with only locally isolated recrystallized coarse grains containing a high gamma prime precipitates density (Figure 12a and 12b). In addition of double grain size population, U720Li disk also show isolated non-recrystallized grains of around $340 \mu\text{m}$ (Figure 13) slightly larger than those observed in AD730™ disks (ALA grain size close to $110 \mu\text{m}$). Isolated unrecrystallized grains come from billet and are mainly localized on the upper rim of the part where the original fibers of the billet are less deformed.

Figure 12: Typical microstructure on the same position for the three materials, on the bore of the disk: Homogeneous microstructure on AD730™ disk a) from small ingot and b) from large ingot - c) Banded microstructures on U720Li disk

Figure 13: a) Isolated non-recrystallized grains around 340 μ m observed on U720Li disk – b) Focus on one unrecrystallized grain on U720Li disk

Gamma prime precipitates were observed and measured on two different positions of the disks. The first position (n°1 Figure 2) is located near the rim and the cooling rate is estimated to be equal to 290 °C/min. The second position (n°2 Figure 2) is located near the core and the cooling rate is estimated to be equal to 174 °C/min. The secondary gamma prime precipitates sizes for the position n°2 are a bit larger (~80nm) than those of the position n°1 (~60 nm) (Figure 15a and b). These results suggest that a slight difference of cooling rate has an impact on gamma prime precipitates. For a same position on the disk, the size of secondary gamma prime precipitates is quite similar for the three materials (Figure 16). However, in comparison with U720Li disk, a higher amount of tertiary gamma prime precipitates was observed on AD730TM disks.

UT inspection results

Ultrasonic Testing (UT) inspection was performed on each disk with the same parameters. The maximum noise level evaluated as an equivalent flat bottom hole (EFBH) was measured on each disk (Figure 14). All the disks made from AD730TM small ingot and AD730TM large ingot show a lower maximum noise level than U720Li disks. These results could be related to the presence of unrecrystallized grains on U720Li disks. We can also notice the homogeneous results obtained on AD730TM disks.

Figure 14: Maximum noise for each disk and material

Figure 15: SEM-FEG examinations on γ precipitates on AD730TM large ingot from a) Position n°1 (cooling rate 290 °C/min - secondary γ precipitates sizes ~60 nm – b) position n°2 (cooling rate 174 °C/min - secondary γ precipitates sizes ~80 nm)

Tensile tests

Ten positions in the disk were tested to obtain reliable information on mechanical properties variation. For each position, the cooling rates during quenching after solution heat-treatment were determined by numerical modelling between 1050 °C and 700 °C. Tensile tests were performed at three different temperatures: room temperature (RT), 650 °C and 720 °C. All the results are given in Table 3. Tensile strength is quite homogeneous into the parts for the three billets with a maximal variation equal to +/-50MPa on ultimate tensile stress (UTS) and +/-30MPa on yield stress (YS). Yield strength measurements ranked from the lowest to the highest cooling rates for the three tests temperatures are plotted on Figure 17. Higher tensile properties at high temperature are noticed for AD730TM in comparison with U720Li, independently of the ingot size. These higher tensile properties could result from the higher amount of tertiary precipitates and also the higher reinforcement by refractory elements of gamma matrix [12]. At high temperature, a higher impact of the cooling rate on YS properties was observed.

Figure 16: SEM-FEG examinations on γ precipitates made on position n°1 for each disk: a) and b) AD730TM small ingot - secondary γ precipitates sizes $\sim 47\text{nm} \pm 9\text{nm}$ - c) and d) AD730TM large ingot secondary γ precipitates sizes $\sim 62\text{nm} \pm 7\text{nm}$ - e) and f) U720Li secondary γ precipitates sizes $\sim 60\text{nm} \pm 11\text{nm}$

		AD730 small ingot			AD730 large ingot			U720Li		
		RT	650°C	720°C	RT	650°C	720°C	RT	650°C	720°C
UTS MPa	Average	1582	1365	1181	1587	1362	1189	1583	1330	1115
	Min	1570	1336	1152	1572	1336	1167	1571	1307	1090
	Max	1600	1429	1215	1605	1392	1234	1591	1351	1130
YS MPa	Average	1247	1113	1096	1247	1116	1097	1113	1048	1010
	Min	1230	1078	1070	1227	1093	1075	1097	1036	1026
	Max	1272	1140	1113	1278	1140	1127	1144	1063	993
E %	Average	23	24	15	21	20	13	22	30	22
	Min	22	19	11	20	17	11	21	22	16
	Max	24	26	21	24	22	15	24	35	31
RA %	Average	25	28	20	20	25	17	21	37	28
	Min	23	21	15	18	20	15	18	28	18
	Max	28	31	27	25	28	20	28	47	38

Table 3: Tensile results on three materials obtained with 10 tests for each part

Figure 17: Tensile tests results on three materials from LPT disks

Creep tests

Four positions located in the bore and in the rim of the disk were tested in different creep conditions. Stress rupture tests performed at 720 °C under a stress equal to 500 MPa exhibit time to rupture exceeding 200h for the three materials (Figure 18). Creep tests performed at 670 °C under a stress equal to 650 MPa confirm that creep properties of AD730TM to be at least as good as those of U720Li (Figure 19). This better creep behavior of AD730TM at 670 °C could result from the higher amount of tertiary precipitates and also from the higher reinforcement by refractory elements of gamma matrix [14] which both contribute to impede dislocations motion.

Figure 18: Stress rupture tests results performed at 720 °C under a stress equal to 500 MPa for the three materials

Figure 19: Creep tests results performed at 670 °C under a stress equal to 650 MPa on three materials from LPT disks

Fatigue tests

Strain controlled LCF tests (600 °C, $R=0$, $\epsilon=0-0.75\%$) were performed on ten specimens cut out from each disk and the nature of all crack initiation sites were identified. Numbers of cycles to failure, ranked from the lowest to the highest cooling rate, are given in Figure 20. Good fatigue results were obtained on AD730™ large ingot disk with fatigue lives above $1E5$ cycles. The equivalent diameter of carbonitrides responsible for fatigue crack initiation was evaluated to be respectively close to 18 μm and 24 μm for disks from AD730™ small ingot and AD730™ large ingot (Figure 21). These results are in good agreement with the measurements made by image analysis on AD730™ billets where the largest carbonitrides for AD730™ small ingot and AD730™ large ingot were measured to be respectively equal to 17 μm and 23 μm (Figure 9). It is noteworthy, that the lowest fatigue results (N_f below $1E5$ cycles) was obtained on disk from 508 mm diameter ingot. Consequently, the largest carbonitrides observed on the part from 635 mm diameter ingot did not lead to lower fatigue properties. LCF properties of AD730™ are quite similar to those of U720Li.

Figure 20: LCF tests results at 600 °C ($\epsilon=0-0.75\%$, $R=0$) on three materials from LPT disks

Figure 21: Example of LCF initiation sites on AD730™ parts: a) from AD730™ small ingot, carbonitride with 18 μm mean diameter ($N_f = 67\,523$) b) from AD730™ large ingot, carbonitride with a 21.9 μm mean diameter ($N_f = 362\,746$)

Summary on LPT disks results

Homogeneous microstructure was obtained on AD730™ disks compared to U720Li disk where isolated non-recrystallized coarse grains were observed. Gamma prime precipitates size is homogeneous on AD730™ disks and a bit finer than those on U720Li disk. All the disks made from AD730™ small and large ingots show a lower maximum noise level than those of U720Li disks. No detrimental effect of larger AD730™ ingot diameter on mechanical properties was observed. Higher tensile properties at high temperature were obtained for AD730™ in comparison with U720Li, independently of the ingot size. AD730™ creep properties are noticed to be at least as good as those of U720Li. Fatigue tests showed that the largest carbonitrides observed on the part from AD730™ large ingot did not lead to lower fatigue properties.

Summary/Conclusions

This work is a reliable demonstration of AD730TM larger ingot feasibility to enhance the combination between cost and properties. No detrimental effect of larger ingots was detected and fatigue properties as well as micro-segregations remain at a good and acceptable level. Mechanical properties of AD730TM and U720Li are very similar. There are even advantages on tensile strength at high temperature and creep elongation for AD730TM.

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