## StereoLithography Epoxy Resin Development: Accuracy and Dimensional Stability

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#### Abstract

Recently, a new class of StereoLithography (SL) resins was developed that provide significantly improved overall part accuracy and dimensional stability relative to earlier SL resins. The new resin formulation, XB 5170, is based on epoxy chemistry and has many substantial advantages over conventional acrylate SL resins. In addition to excellent mechanical properties, the epoxy resin has very low shrinkage, resulting in extremely low curl and distortion. A standard UserPart built in XB 5170 achieved the highest level of dimensional accuracy to date from a statistically significant number of measurements taken in the x, y, and z directions. SL parts built in XB 5170 also exhibit superb dimensional stability in the laser-cured state as demonstrated by a new creep test. Dimensional stability in the laser-cured state is critical, especially for parts built in the new QuickCast<sup>TM</sup> build style. In addition to the epoxy resin, QuickCast is the key to successfully utilizing SL parts for direct shell investment casting applications.

#### Background

The StereoLithography (SL) process involves building 3-dimensional objects by sequentially generating thin layers of selectively cured liquid resin on top of each other. The current StereoLithography Apparatus (SLA) uses a focused UV laser beam directed by a computer-controlled X-Y scanning mirror system that has an extremely high pointing accuracy. High laser pointing accuracy is undoubtedly a prerequisite to generating an accurate part. However, high pointing accuracy alone does not automatically translate into building accurate parts. Many variables such as resin type, laser spot size and symmetry, and the process used to build a part also affect part accuracy. Therefore, the accuracy of final SLA parts, in addition to inherent machine accuracy, is very important to SL users.

The process parameters required to build accurate SL parts are quite complex in nature. However, insights obtained from fundamental research at 3D Systems in 1990<sup>1</sup> led to a substantial improvement in SL part accuracy. WEAVE m and STAR-WEAVE m laser drawing styles developed in 1990 and 1991, respectively, increased the accuracy of acrylate resin by more than 40% based on a statistically significant number of measurements taken on a standard accuracy part called the UserPart.

The SL UserPart was designed, not by 3D Systems, but by the SLA user group in 1990 to determine the overall dimensional accuracy of SL parts.<sup>2</sup> From a significant number of UserPart measurements accumulated since then, SL has achieved a relatively high level of accuracy even in

the conventional acrylate resins.<sup>3</sup> However, it appeared as if the accuracy of SL parts made in acrylate resins and the process optimization techniques were approaching a plateau.

Substantial improvements in part accuracy and resin properties were necessary to apply SL to new markets beyond conventional form, fit, and function prototypes. For instance, a higher level of accuracy is necessary to expand the applications of SLA into the manufacturing arena.

Dimensional stability is also essential for a rapid prototyping part for almost any application. Without sufficient dimensional stability even a part initially built accurately becomes inaccurate over time. A rapid prototyping part would have limited use without high accuracy sustained for a period of time characteristic of the application. For form, fit, and function applications, dimensionally unstable parts may not fit properly. Holes and other intricate features may become misaligned with time.

A high level of dimensional stability becomes especially crucial in an application such as direct shell investment casting. This process requires an efficient conversion of prototype parts from plastic to metal with maximum preservation of accuracy. In shell investment casting, a rapid prototyping part is normally subjected to periods of days up to even weeks before it is shelled. Only rapid prototyping sites with an in-house foundry are capable of initial face-coat shelling within hours after the parts are built. Otherwise, a nontrivial amount of time, typically on the order of days, is required for shipping the part to an appropriate foundry and for the foundry to actually shell the part. Any dimensional instability within this period leads to an inaccurate shell investment casting pattern, rendering the resulting metal cast prototype unacceptable. Clearly, a new type of resin with high accuracy and excellent dimensional stability was needed.

## New Epoxy Resin XB 5170

Ciba-Geigy (Switzerland) and the Research and Development Department at 3D Systems have been involved in a program of joint SL photopolymer research and development since 1988. Ciba-Geigy, one of the leading chemical companies in the area of epoxy chemistry, had been looking at SL epoxy resins for sometime.

In July, 1993, Ciba-Geigy released a new resin for the SLA-250 based on epoxy chemistry. The characteristics of this epoxy formulation, XB 5170, are given in table 1. The properties of two conventional SL acrylate resins are also listed for comparison. StereoLithographic properties of the epoxy resin, XB 5170, compare well with those of acrylate resins except that the photospeed is a little slower. The epoxy resin has an excellent set of physical properties that exceed those of acrylate resins in almost every single category. Namely, XB 5170 has:

- 1) very low viscosity
- 2) high laser-cured (green) modulus
- 3) high postcured modulus
- 4) good elongation at break
- 5) good impact strength

However, the high achievements in accuracy and dimensional stability are what makes this new epoxy resin so special. According to the diagnostic accuracy measurements performed, XB 5170 shows:

- 6) substantially reduced thin flat slab distortion
- 7) almost twice the overall UserPart accuracy compared to XB 5081-1 acrylate resin (Note: XB 5081-1 was formerly the most accurate SL resin until XB 5170 was introduced.)
- 8) negligible Cantilever curl during the SL building process.
- 9) superb dimensional stability as measured by the CreepBar diagnostic test.

In addition, the epoxy resin, when built in the new QuickCast <sup>TM</sup> build style, is especially suited for direct shell investment casting applications.<sup>4</sup> Many metal parts with high accuracy and smooth surfaces have been cast successfully in the epoxy resin, XB 5170.<sup>5</sup>

Even though the physical properties of XB 5170 are important and definitely deserve discussion of their own, this report will focus on the first three diagnostic tests given in Table 1, that determine the accuracy and dimensional stability of the epoxy resin.

Resin Type	Acrylate	Urethane Acrylate	Epoxy
Resin Name	XB 5081-1	XB 5149	XB 5170
	<b>Dimensional Prope</b>	erties and Accuracy	
UserPart Accuracy* (R.M.S. Error)	5.3 mils	6.1 mils	2.8 mils
Cantilever Curl*	10 %	10 %	1%
Green Creep Rate*	3.7 mil/log <sub>10</sub> t	11.5 mil/log <sub>10</sub> t	0.5 mil/log <sub>10</sub>
Linear Shrinkage	0.7 %	0.6 %	0.06 %
Flat Slab Distortion	71 mils	70 mils	20 mils
	<b>Physical</b>	Properties	
Viscosity @ 30°C	2,400 cps	2,000 cps	180 cps
Green Flexural Modulus**	620 MPa	310 MPa	1700 MPa
Cured Tensile Modulus***	3,000 MPa	1,150 MPa	2,700 MPa
Elongation@Break***	2.5 %	10 %	9 %
Impact Strength***	3 kJ/m²	23 kJ/m²	30 kJ/m <sup>2</sup>
	StereoLithogra	phic Properties	
Critical Exposure, Ec	7 mJ/cm <sup>2</sup>	6 mJ/cm <sup>2</sup>	9 mJ/cm <sup>2</sup>
Penetration Depth, Dp	7 mils	5.5 mils	4.5 mils

\* Discussed in Text.

\*\* 2.5 mm Thick strip built on SLA.

\*\*\* Specimens cast between glass & UV cured.

## Table 1.

#### UserPart, Cantilever, and CreepBar Accuracy Diagnostic Parts

Diagnostic tests are essential tools used to compare the achievable level of performance for new resin systems or new process techniques. Diagnostic tests allow one to compare new resin or process systems with earlier ones in a quantitative manner. The dimensional properties of the epoxy SL resin, XB 5170, were determined by the following diagnostic tests.

Three diagnostic test parts, UserPart, Cantilever, and CreepBar, shown in figure 1, are representative diagnostic test parts used to determine dimensional properties. The UserPart and the Cantilever parts, discussed in detail elsewhere<sup>6,7</sup> are a measure of overall dimensional accuracy, and curl distortion during the building process, respectively. The CreepBar test was recently developed specifically to determine the dimensional stability of an SL part over a given period of time.



Figure 1. Three accuracy diagnostic parts used to test StereoLithography resins.

#### **UserPart Accuracy**

The UserPart is intended as a metric of overall SL part accuracy. It is 9.5 inches long by 9.5 inches wide and about 2 inches high. Many small and large features are included. The accuracy data is obtained by measuring a statistically significant number of dimensions of the features that have varying thicknesses and lengths. From a single UserPart, 78 measurements are taken in the x and y directions and 14 measurements are taken in z. The total number of dimensional measurements per part is 170. These dimensions include thin, medium, and thick walls, short and long dimensions, as well as holes of various sizes. All measurements are taken using a Coordinate Measuring Machine (CMM) that has a repeatability of  $\pm 5$  microns, or  $\pm 0.2$ 

mils. The accuracy data obtained from the UserPart includes the normal shrinkage of the resin that takes place in the vat during the SL building process. It also takes into account the dimensional changes that occur during the postcuring process. In other words, the UserPart is subjected to all of those processes incurred when building real SL parts.

To analyze overall part accuracy, the measured UserPart dimensions are compared to the nominal CAD dimensions and dimensional error values are calculated. A histogram called an Error Distribution Function is then constructed from compiling individual error values. The Error Distribution Function for the conventional acrylate SL resin, XB 5081-1, is given in figure 2. The curve is a result of 2550 total data points obtained from 15 UserParts all measured by CMM. The peak occurs at error=0 and the distribution curve is quite symmetrical. The fact that the general shape resembles a Gaussian distribution suggests that the errors on the plot are random events and are not due to systematic errors.

The overall dimensional accuracy of the SL UserPart built in XB 5081-1 can be extracted from the error distribution curve. However, it is usually more convenient to convert the curve into a Cumulative Error Distribution, as shown in figure 3. This is done by simply flipping all the data points in the negative error region to the positive error region and taking a running sum at each specified error window. Using the Cumulative Error Distribution the probability that any dimension will lie within a certain range of error can be immediately interpolated. For example, a commonly quoted characteristic error value is the root-mean-square (R.M.S.) error or equivalently, the standard deviation. The R.M.S. error corresponds to a 68% probability of occurrence.

For XB 5081-1 resin, the R.M.S. dimensional error is  $\pm 135$  microns or  $\pm 5.3$  mils. This means that 68% of the dimensional measurements taken on the UserPart made in XB 5081-1 are expected to lie within  $\pm 5.3$  mils. XB 5081-1 was formerly the most accurate SL resin, until the epoxy resin was recently introduced.

Now, what about the new epoxy resin XB 5170? The Error Distribution Function for a UserPart built in XB 5170 is given in figure 4. Because this resin is just being released, the curve was generated from a single UserPart. Nevertheless, the error distribution curve generated from 170 dimensional measurements already looks quite Gaussian. The peak of the curve is also centered at about error=0. The Cumulative Error Distribution for XB 5170 is plotted in figure 5.

It is clear that the error for the epoxy resin is a lot less than XB 5081-1. The maximum error at 100% probability, is about 500 microns or 20 mils for XB 5081-1, compared to less than 355 microns or 14 mils for the new epoxy resin, XB 5170. The R.M.S. error for XB 5170 is only 71 microns or 2.8 mils. XB 5170 resin provides almost twice the overall accuracy. It should be appreciated that the degree of improvement in the R.M.S. error from 5.3 mils to 2.8 mils, based upon a statistical number of data points, is truly significant.

A history of progress in SL UserPart accuracy is shown in figure 6. In 1989 the R.M.S. error for a UserPart built in the original 50-mil Tri-Hatch build style was about  $\pm 9$  mils. With the introduction of WEAVE  $\pm$  and STAR-WEAVE  $\pm$  build styles in 1990 and 1991, and subsequent optimization in 1992, the R.M.S. error was reduced by a factor of two to  $\pm 4.5$  mils using the same resin. This was comparable to the accuracy achieved by a UserPart machined from an engineering plastic (Perspex) by a CNC milling machine.<sup>8</sup> This level of accuracy is marked by the horizontal line in figure 6. With the introduction of the epoxy resin, XB 5170, the UserPart R.M.S. error was dramatically reduced to  $\pm 2.8$  mils. This improvement brings the accuracy of SL generated UserParts into the same ballpark as an Aluminum UserPart machined by CNC milling<sup>9</sup>, a significant advance in the accuracy of SL parts.



Figure 2. Error Distribution Function for fifteen UserParts made in XB 5081-1 resin.





Cumulative Error Distribution for fifteen UserParts made in XB 5081-1 resin.











Figure 6. StereoLithography's progress in UserPart Accuracy from 1989 to 1993.

#### **Unsupported Cantilever Curl**

The Cantilever diagnostic test, shown in Figure 7, tells us how much the protruding Cantilever areas of a part curl if they are not properly supported. Therefore, cantilever curl is one type of distortion that happens in the vat during the building process. This was a useful measure in the past when feasibility studies were performed to see whether or not parts could be built with minimal supports. Today, cantilever sections of real SL parts are usually supported such that negligible cantilever sections occur. Therefore, while the Cantilever diagnostic part is still very useful in comparing different resin systems, it has some limitations in comparing real parts built with proper supports.

Nonetheless, Cantilever curl is indeed a measure of the inherent tendency for the resin to undergo warpage during the building process. The curl values (rise at 6-mm run) for the conventional acrylate resins manufactured by Ciba-Geigy are typically between 8~13%. As a specific example, for XB 5081-1 it is 10%.

However, the Cantilever curl for the epoxy resin, XB 5170, is extremely small. The curl value for XB 5170 is only 1 %. This result indicates that the epoxy resin has a very small, almost negligible tendency to undergo curl distortion during the building process.



Figure 7. Cantilever free (unsupported) curl phenomenon.

## **CreepBar Creep Distortion**

So what types of accuracy problems do the supported parts typically have? Experience with the conventional acrylate resins has shown that when SL parts are left in the laser-cured or so-called "green" state for a long time without postcuring, their dimensional errors increase. The longer they stay in the laser-cured state, the more inaccurate the parts become. This indicated that SL parts built in acrylate resins are not completely dimensional stable in the laser-cured state.

For visualization purposes, the degree of dimensional change with time for SL parts is relatively small and causes no problems. However, dimensional instability and its time dependence must be identified and understood in order to build highly accurate and more dimensionally stable parts that meet the requirements for direct shell investment casting applications. Furthermore, resins and specific build processes must be found that can indeed generate parts with high dimensional stability.

While layer-addition fabrication offers many advantages such as the ability to build complex geometries; curl distortion has traditionally been one of the major disadvantages. When additional layers are cured or solidified on top of each other, physical or chemical transformation takes place in the material. For SL, it is a photochemical crosslinking reaction, and for rapid prototyping methods that use solidification of a molten material, it is changes in the density of the materials due to temperature gradients. Both of these changes involve some degree of volumetric shrinkage and lead to the build up of internal stress in the part. This built-in internal stress ultimately manifests itself in a type of warpage known as creep distortion.

Creep distortion may take place quickly or slowly with time. The exact creep behavior depends on many parameters including the part geometry, type of resin and its laser-cured glass transition temperature, ambient temperature, and build parameters. Creep distortion becomes worse for parts having flat geometries and high aspect ratios when the long axis coincides with the building surface. In this report, time dependent creep distortion measurements are presented for a number of SL resins. The CreepBar is a new diagnostic test that was designed to measure the dimensional stability of an SL part. In the CreepBar diagnostic test, a thin rectangular part, shown in figure 8, having a high aspect ratio, is built fully supported on an SLA. The supports prevent any distortion from occurring in the vat. The CreepBar is then taken off the platform, cleaned, and is allowed to undergo deformation with time. The CreepBar is laid such that the direction of creep distortion is horizontal, to minimize the effect of gravity. An optical creep measurement (OCM) device, shown in figure 9, is used to track the rate of creep distortion with time. The data is recorded on a computer. The resulting creep distortion is a quantitative measure of the dimensional stability of the test part, for a particular resin built in a particular way.

Laser-cured and postcured CreepBar behaviors are expected to be very different. They should be clearly distinguished from each other. For most resin systems, the great majority of the creep distortion occurs in the laser-cured state. Therefore, only laser-cured CreepBar data was considered at this time. The CreepBar, thus, is NOT postcured for the data presented here.

Laser-cured creep distortion arises from built-in stresses generated during the building process on the SLA. Postcured creep distortion is more complex because it involves stresses that are introduced into the part during UV or thermal postcuring in addition to the original laser-induced stresses. Both classes of creep distortion depend strongly on the geometry of the part, on the resin, and the SL build parameters used, as well as temperature. The experimental results for creep distortion should be compared only for the creep tests performed under a controlled environment.

It should be noted that the creep test presented here is distinctively different from the ASTM creep test that involves an externally applied standard weight as a source of stress. In the SL creep test, the internal build stresses serve as the distortion mechanism, and may not be constant throughout the creep phenomena.



# Figure 8. CreepBar diagnostic part dimensions and a schematic of the test sequence and the creep phenomenon.



## **Creep Experiment**

Creep Distortion is a deformation of an SL part that takes place with time. It is the degree of deformation that happens after the SL part is taken off the platform and the supports are removed. The deformation does not begin until this instant because the supports and the platform restrict the SL part from deforming. The built-in stresses begin to affect the dimensional stability of the SL part only after these restrictions are relieved.

In the creep test a high aspect ratio  $(8 \times 1/4 \times 1/4 \text{ inch})$  rectangular bar called the CreepBar, is built on an SLA. It is supported completely during the building process in the SLA to prevent it from distorting in the vat. It is then removed from the SLA platform. The degree of out-of-plane curl distortion of the laser-cured bar, marked by the small downward arrow shown at the bottom of figure 8, is measured over a period of 24 hours.

A CreepBar of length = 200mm, width = 6.35mm, and height = 6.35mm is built on an SLA, as shown in figure 8, with supports such that the part does not undergo distortion during the building process. When the CreepBar is complete, it is rinsed in TPM (no solvent cleaning is required for the QuickCast m build style) and then is taken off the platform with the supports removed as soon as possible. The time that the CreepBar is taken off the platform is taken as time t = 0.

As shown in figure 8, the CreepBar begins to undergo creep distortion with time. Because of the way the layers are cured in the SL process, the direction of the stresses force the CreepBar to distort concave upward. This may be thought of as a "latent curl" effect that manifests itself only after the supports are removed.

As soon as the part is cleaned and removed from the supports, it is placed on a custombuilt optical device called the OCM (Optical Creep Measurement). An optical approach was elected because mechanical means of measurement for small displacement distances are both tedious and unreliable. Even a small mechanical force applied to the CreepBar could distort it. A schematic drawing of the OCM device is given in figure 9. The OCM consists of flat supporting plates that are co-linear, a photo detector, a small section of Aluminum foil attached to the CreepBar, and an IR photodiode light source shining down on the photodetector. (The light source is not shown in the schematic drawing.) The IR photodiode does not initiate further photopolymerization.

The principle of OCM operation depends on the variation in total optical power received by the photodetector as the result of displacement by the CreepBar. The voltage reading from the photodetector depends on the total amount of incident light. At time=0, when the CreepBar has not undergone any distortion, the voltage reading is low because most of the IR light is blocked by the CreepBar and the Aluminum foil.

After some elapsed time, the CreepBar distorts and the aluminum foil moves away from the photodetector. A larger area of the photodetector is now exposed to the light source and the voltage reading increases accordingly. From a carefully constructed calibration curve, shown in figure 10, the voltage reading can be directly converted into creep distortion. The creep distortion data is then collected as a function of elapsed time on the computer.



Figure 10.

Calibration curve for the Optical Creep Measurement Device relating voltage output to creep distortion.



Figure 11. Creep distortion of laser-cured SL resins as a function of time at 21°C.





Creep distortion data plotted as a function of the logarithm of time at 21°C. The plot shows every fifth data point and best-fit straight lines.

#### Creep Data

Green Creep Data for acrylate resins, XB 5143, XB 5149, XB 5081-1, and the new epoxy resin XB 5170 were collected on the OCM. All creep measurements were taken at room temperature ranging from 20 to 22°C, typically at 21°C. The results are presented on figure 11. All creep data show a rapid increase in creep distortion at short times. The creep data appear to reach a plateau at long times. For most of the resins presented here, more than 60% of the 24-hour creep distortion is virtually complete within an initial period of 2 hours.

With respect to absolute magnitude, CreepBars made in XB 5143 have the largest creep distortion. They distort to about 50 mils/8-inch length in 24 hours. This suggests that *laser-cured parts should be postcured as soon as possible in order to preserve accuracy*. This precaution is usually taken by most SL users. XB 5143 is then followed by XB 5149, XB 5081-1 and XB 5170. XB 5081-1 shows a dimensional instability of about 15 mils/8 inches over 24 hours.

However, the new epoxy resin, XB 5170, has the least creep distortion at every point in time. This creep data shows that the epoxy resin is, by far, the most dimensionally stable resin in the laser-cured state. At every point in time, the creep distortion for XB 5170 is less than 5 mils/ 8-inch length over a period of 24 hours.

Because the rate of creep distortion was found to be rapidly decelerating with time, the creep data was plotted as a function of the logarithm of time in figure 12. Interestingly, all of the creep data was found to be very nearly a log-linear function of time in the 24-hour period. Note the linearity of the data in figure 12. From this observation, a single useful parameter called "creep rate" could be defined for purposes of comparing the creep properties of various SL resins.

Creep rate, defined by the symbol CR, is basically the slope of the creep data when it is plotted as a function of the logarithm of time. Thus, CR is the creep distortion that takes place for every multiple of 10 in time.

For example, consider an SL resin CreepBar that was built in a particular build style and was found to have CR = 15 mils. As the elapsed time increases from 1 hour to 10 hours (one multiple of 10), the creep distortion would increase by 15 mils. As the elapsed time then increases from 10 hours to 100 hours (another multiple of 10), the creep distortion would increase by an additional 15 mils. The total distortion, as time elapses from 1 to 100 hours (two multiples of 10 in time), is twice the CR, or 30 mils. Thus, a single parameter, CR, or creep rate, characterizes the dimensional stability of SL parts.

A summary of CR values for SL resins is presented in table 2.

SL Resin	CR	
	(mils / $\log_{10}$ time)	
XB 5170	0.5 mils	
XB 5081-1 3.7 mils		
XB 5149 11.5 mils		
XB 5143	14.4 mils	

#### **Creep Rates of StereoLithography Resins**

## Table 2.

It is important to remember, though, that SL parts do not continue to creep indefinitely even in the laser-cured state. Internal stress is relieved as the parts undergo creep. When it becomes negligibly small, an equilibrium is reached. Once a distorted equilibrium position is attained the part no longer creeps. The rate at which equilibrium is reached depends on the resin and involves photochemical, process, and viscoelastic parameters, as well as temperature. Also, when SL parts are postcured, their creep rates decrease tremendously.

Nevertheless, it is clear, from the CreepBar data shown above, that the new epoxy resin, XB 5170, shows extremely low creep. Consequently, SL parts made from XB 5170 exhibit superior dimensional stability in the laser-cured state.

## Conclusion

A StereoLithography epoxy resin, XB 5170, intended for use in the SLA-250 system was recently released by Ciba-Geigy. This resin has excellent physical properties and outstanding dimensional properties. Namely, it is highly accurate and dimensionally stable.

The dimensional properties of this epoxy resin were presented in comparison to the conventional SL acrylate resins. Accuracy and dimensional stability values were obtained from three diagnostic tests called the UserPart, Cantilever, and CreepBar.

The UserPart showed that *dimensional accuracy for XB 5170 was almost twice as good as the next best acrylate resin.* This SL UserPart was found to have the dimensional accuracy comparable to a UserPart built in aluminum by a CNC milling machine.<sup>8</sup>

The Cantilever curl diagnostic test showed that XB 5170 has a negligible tendency to curl during the SLA building process. For conventional acrylate resin, the best curl value was about 7% curl. XB 5170 curled only 1%, which is an improvement of a factor of seven, and indicates that negligible internal stress is introduced into parts when they are made in this epoxy resin.

A new diagnostic test was introduced to demonstrate dimensional stability of SL parts. The test, called CreepBar distortion test, involved the measurement of the out-of-plane creep distortion of an 8-inch long bar built on an SLA. This test also showed that XB 5170 undergoes minimal creep distortion in the laser-cured state. According to the CreepBar test, XB 5170 is at least 7 times more dimensionally stable than XB 5081-1, which itself had been historically known as a relatively dimensionally stable SL resin.

An improved level of accuracy and dimensional stability was achieved by the new epoxy resin, XB 5170. The diagnostic tests showed superb overall accuracy, low cantilever curl, and very high dimensional stability. These properties, together with outstanding physical properties such as low viscosity and high green strength, make XB 5170 the resin of choice for StereoLithography, especially when high accuracy is required.

An additional advantage is that SL parts built in the new QuickCast <sup>™</sup> build style with XB 5170, have been proven to be effective in direct shell investment casting applications.<sup>4,5</sup> Therefore, SL users can expect XB 5170 parts to have greatly improved accuracy and dimensional stability as well as direct shell investment casting capability.

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