

Melting and Refining of Superalloys and Titanium Alloys

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(Received on November 12, 1991; accepted in final form on December 20, 1992)

The processes used for the manufacture of both superalloys and titanium for the aerospace industry *i.e.* vacuum induction melting, vacuum arc melting and electroslag remelting, are to be considered as mature technologies. They have been developed over many years in regard to both the equipment used and the process techniques. They have been found to have advantages and have been manifestly successful in producing high quality material as attested to by the impressive reliability of turbines in service. However, in the present progress of turbine manufacture we have arrived at a position where the operational improvements in engine performance require alloy components of even higher reliability—beyond that which can be guaranteed by the present processes, for reasons which are discussed below. We are hence at an interesting stage in the development of the melting processes where we have little potential left in the present techniques and must therefore introduce the next stage in the technical development. The discussion below outlines the rationale for the choice of this type of new process, based on both absolute quality and also on quality assurance through process control. It is concluded that for both titanium alloys and superalloys, electron-beam melting can provide the incremental improvement which we need, as is being presently demonstrated by the industrial introduction of electron beam technology.

KEY WORDS: superalloys; titanium alloys; turbine materials; vacuum melting; electroslag melting; electron beam melting.

1. Introduction

The manufacture of components for aero-engine and gas turbine use is entering an interesting phase, in which we can see clear limitations in the present range of materials and also clear requirements for the future designs. The design philosophy is beginning to make a significant impact on materials manufacture. We have no new materials in realistic prospect which could provide a substantial increase in, for example, operating temperature and it is beginning to be more widely recognized that the economic key to turbine advance is not materials properties but, instead, is materials reliability. We have, for example, a perceived ultimate limit in turbine inlet temperature of around 1650°C in conventional air-breathing machines, due to limits in NOX exhaust production, which imposes in turn, clear ultimate limits on what might be expected from the use of higher temperature materials. On the other hand, in spite of several decades of development, the present most-widely used turbine disk alloy (IN718) is limited to less than one-fifth of its theoretical fatigue life because we cannot yet guarantee the absence of random defects in its manufacture. The impact on the turbine's overall economic efficiency of extended component life is more important than increases in instantaneous output or thermodynamic efficiency gained at the expense of reliability. This maxim holds true for all turbine

applications, in civil or military aircraft engines and in power turbines.

The above finding also has an important bearing on engine safety. At present our designs are heavily based on a statistical prediction of probable defect distribution, which is supported by NDT inspection. It results in the conservative life-cycle predictions and retirements cited above. Although the industry has a remarkable record of safety and success with these techniques, as we bring operating requirements closer to the material's limits, we can anticipate that failures will occur more frequently and that testing techniques will become less reliable. Obviously, rather than to put the producer into the philosophically-impossible position of guaranteeing the absence of defects which he cannot find, it would be more desirable to have a manufacturing process which did not produce the defects in the first place even at a higher cost. With that objective we remove the need for the statistics-based fracture analysis and the concomitant uncertainty in life-cycle prediction. This approach has been termed "Zero Defect" manufacturing and is the goal of superalloy and titanium manufacturers worldwide.

The various processes and process combinations presently used by the industry constitute a mature technology, with well-defined limits of operation and well-developed equipment. It is unlikely to contain unknowns which would substantially change those limits.

In the turbine industry, the materials and manufacturing methods are uniquely restricted by specifications and agreements into frozen practices. This situation causes the industry to progress in step-increments rather than on a continuous basis. It tends to store good ideas until the opportunity for change occurs and then to make relatively-large changes. Such a change is presently taking place, driven by new turbine design with new user-requirements.

2. Industry Present Practices

The industry standards for superalloy manufacture centre around VIM, VAR and ESR; those of titanium around VAR only. These processes have been amply described in the literature.¹⁻⁴⁾

2.1. Vacuum Induction Melting

There is no practical alternative to the VIM process for obtaining the close chemical control and homogeneity required by specification. The complete melting and mixing of the charge components is essential to obtain the required constant compositions. Unfortunately, the complete melting also requires the use of a ceramic crucible, which produces a characteristic range of problems. The principal problem is that of metal/refractory reactions leading to crucible disintegration producing agglomerated oxide inclusion particles in the liquid metal charge. The problem can be minimized by careful choice of refractory where possible using prefired shapes and also by using the minimum of jointing compounds; by keeping the system under vacuum or argon to the maximum extent; and by reducing the possibility for refractory degradation by thermal shock or overheating. However, even in the best of circumstances, the refractory attack will still take place and we must use "add-on" procedures to attempt inclusion removal.

The simplest of these methods is the use of special tundish designs which are intended to form a quiescent pool during the pouring process, hence allowing inclusions to separate. This method is also generally combined with the use of ceramic filters⁵⁾ to further separate the inclusions. This system has been shown to offer advantages and is widely practiced in the casting industry, but has several drawbacks. First, the filters used offer significant flow resistance and the consequent heat loss tempts the melter into providing a compensating additional superheat. The higher temperature prevents filter clogging, but also defeats the filter purpose by solutioning many of the inclusions we wish to remove, particularly TiN. Tundishes with the heating necessary to solve this dilemma have so far not been used in commercial practice. The second major disadvantage in filter use lies in the necessary quality control problem. At the end of the process, the filter is full of solid metal and it is difficult, if not impossible, to certify both its integrity and the fact that it indeed acted as a filter. The possibility of undetected breakage remains a worrisome prospect for process control.

The VIM process generally has provided high-quality alloys for many years, but as the high rejection-rate in

critical castings attests, it has quite definite limitations in output quality. Added to this finding is the fact that it is a process containing too many operator-sensitive functions for modern process control. The result of this situation is that we must regard it simply as a convenient melting/homogenizing step. The alloy will require further refining by more-controlled steps to ensure adequate quality. The starting point for those steps is the VIM casting of electrode shapes.

VAR experience holds that problems in VAR ingots are almost always the result of VIM electrode defects. This generalization is, of course, not completely accurate but has a large element of truth. The VIM electrode is a difficult shape to cast and, despite many attempts to improve the internal quality by VIM process variations such as hot-topping, low-pouring superheat, *etc.*, invariably contains cracks, voids, pipe and segregation. As will be seen below, all of these factors have an adverse effect on the VAR process and can lead to VAR ingot defects.

2.2. Vacuum Arc Remelting

VAR is applied to the melting of both superalloys and titanium, but whilst the equipment for both has superficial similarities, in reality the two applications represent two quite distinct technologies.

In superalloys, VAR is used for the primary purpose of solidification control.⁶⁾ It uses the distinctive VAR heat balance to reduce the local solidification time for the alloy ingot to the point at which the solidification structure is acceptable.⁷⁾ It should be borne in mind that the superalloys have properties which are very largely determined by the primary solidification precipitation and which are difficult to change by subsequent solid-state processing. The interdendritic segregation leads to the precipitation of phases at a eutectic, or succession of eutectics, which can be deleterious to mechanical properties. Examples of these would be Laves phase, primary carbides or primary borides. Since these are the result of eutectic solidification, unreasonably long times are required to decompose them at any temperature below the solidus, and further, such homogenization procedures would involve a finite risk of grain-boundary melting. The objective, therefore, of the VAR solidification control is to assure that the solidification conditions at all positions in the ingot are such that the structure can be homogenized to the point at which it is acceptable for forging operations. The way in which this may be done, using a combination of thermal modelling and VAR process parameters has been described in the literature.⁸⁾ It fundamentally involves the close control of the VAR heat balance through the melting rate and the use of helium injection to cool the ingot surfaces.

The melting of titanium and its alloys has the same basic objective as the processing of superalloys insofar as ingot structure is concerned, with the exception that all titanium systems freeze as single-phase β structures and contain neither inclusions nor primary precipitates in normal practice. Solidification problems are almost invariably those of macrosegregation,⁹⁾ and although little is presently known of the exact form of the

solidification process in titanium alloys, it is likely that the conventional view of dendritic solidification is incorrect at least for CP titanium and the alloy 6Al4V in which a cellular solidifying interface is probably closer to the real situation.¹⁰⁾ The macrosegregation is, however, controlled in the same basic way as is microsegregation for the superalloys, *i.e.*, by controlling the VAR heat balance through the melting rate. Typically, melting rates are at least twice as high for titanium alloys as for equivalent diameter superalloys, and in the case of CPTi and 6Al4V may be five times higher.¹¹⁾ This condition is imposed by the need to have a high temperature in the ingot close to the crucible wall, hence giving the good surface quality necessary for adequate forging yield. The high melting rates result in an ingot temperature profile which is quite different to the superalloy case, leading to a very deep liquid pool.¹²⁾

The contrast between VAR practices for Ti and superalloys may also be seen in the arc conditions applied. In superalloys, the arc is maintained at a very short length (4–6 mm), and in a rapidly-moving condition by the use of a control system based on the frequency and duration of drip shorts.¹³⁾ Stable, constricted arcs give rise to local current concentrations which cause fluid flow in the metal pool and are known to initiate solidification defects arising from flow, such as “freckle.” Early attempts to compensate for this type of flow (and for flow arising from $B \times J$ forces originating in magnetic field imbalance in furnace design) by superimposing a field on the liquid metal using a solenoid winding on the crucible proved to be failures and subsequent development has concentrated on minimizing liquid metal movement by arc control and by eliminating stray fields due to furnace design.¹⁴⁾

With very few exceptions, Ti alloys do not form solidification defects due to metal flow in the ingot pool. Indeed, a judicious choice of flow created by a deliberately-imposed field is used to minimize the macrosegregation problem. In addition, we find that the arc form used for superalloys is not attainable in titanium due to the particular combination of liquid metal physical properties, temperature and current density. Instead, we observe a much longer arc (30–40 mm) which will locate in one position for long time intervals (1–5 sec) unless it is deliberately moved by an imposed magnetic field. We therefore use such a field as an intrinsic part of the control of the process heat balance.

The large ingot pool in Ti VAR contains some of the same features as that for superalloys. Probably the most important feature is that the temperature profiles are largely controlled by the enthalpy input of the liquid metal falling from the electrode. However, even at the high mass flow rates of Ti, most of the pool has little superheat. As is the case with the superalloys, this feature creates the most serious drawback of the process, namely, its inability to dissolve, or melt, particles which fall from the electrode during melting. In the superalloy field, the resulting defect structure of un-melted dendrite clumps¹⁵⁾ is known as “white spot”, and in titanium the same process produces undissolved nitrogen- and/or oxygen-rich areas known¹⁶⁾ as “hard- α .” The reasons why these

defects arise, their structure and effect are quite different, but the basic reason for their existence is contained in the similar ingot thermal profiles.

The form of the melting region in VAR contains many features of interest. The first to note is that the electrode responds to an increased energy input by melting faster, not by increasing the general superheat of metal falling from its melting surface. The liquid metal film on the electrode is very thin (< 1 mm) and remains so even at high melting rates. Local increase in superheat is seen at the metal droplet sites, which have a direct temperature dependence on melting current density. One result of this can be seen in the fact that the incidence of both white spot and hard- α increases with increasing ingot diameter, *i.e.*, with decreasing electrode current density. A second effect is that the electrode acts as a radiation shield for the ingot surface and so plays an important part in determining the ingot heat balance, particularly during the hot-topping period. This effect becomes critical in Ti processing where the arc-gap is large, since any loss of control leading to an increased gap reduces the metal pool temperature at the ingot sidewall and hence creates surface defects. A third point of note is that the electrode also acts as a resistance to vapour transport from the metal surfaces. Although many of the systems we remelt by VAR contain volatile elements (Cr, Mg in superalloys; Mn in steels; Al, Sn and Cr in Ti alloys), when the arc-gap is maintained at a small value, this volatilization can be kept inside the required limits of composition control.

Because arc-gap control is so critical to the success of the VAR operation, anything which perturbs its stability can introduce process defects. The most common source of such problems is the electrode itself. In superalloy melting, the VIM-cast electrode contains porosity, cracks and pipe. The porosity contains sufficient gas (usually CO evolved during solidification) to alter the arc plasma and so disturb the control system. Thermal cracks in the electrodes are also very common, even in systems which employ hot-stripping from VIM and also stress relieving. The cracks act as a barrier to axial heat flow during electrode melting and give rise to characteristic cyclic melt-rate fluctuations during melting. Severe cracking can also provide surface for preferential arc-location and hence disturb the arc-gap control. The electrode shape is a particularly difficult one from the viewpoint of creating a solid shape by casting. Essentially all VIM electrodes contain pipe cavities which will disturb the arc-gap control by both of the above mechanisms. In addition, the pipe is usually associated with the dendrite clumps which form white spots, and also with entrapped slag or inclusion clusters. These latter will also disturb the arc since they change the local thermionic emission characteristics. It is noteworthy that concurrent with the above effects which change arc behaviour, we also have the potential for introducing material into the metal pool (electrode pieces, dendrite clumps, slag, inclusion clusters) under the unfortunate optimum conditions for it to be included into the freezing interface, *i.e.*, whilst the arc-gap control is not operating correctly. For all of the above reason we find, as a generalization, that VAR ingot defects are usually connected with low-quality

electrodes in superalloy melting. As a result, we have developed the practice of interposing an ESR melt between VIM and VAR to form the "triple-melt" process. The primary purpose of the ESR melt is to form an ingot which will constitute the most stable electrode for VAR. This technique is fast becoming the industry standard for all turbine rotating parts. In a sense, it is very similar in intention, although obviously not in detail, to the triple-melting VAR practice for the melting of Ti-alloys for turbine rotating parts.

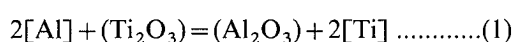
It is clear that the VAR method is a mature technology, with advantages and limitations which are reasonably well understood. In many respects the process has well-served the turbine industry for many years. However, in an environment where we are imposing quality assurance limits which are so close to the ultimate potential of the process, we must question the future potential of VAR in the turbine materials. The maximum diameter of ingot in any given alloy, hence, the available pieceweight is already known or is calculable from solidification data. The statistical frequency with which we can expect to find white spots, or hard- α , is also quite well-defined and is not zero. The inclusion size distributions, primary structure control, *etc.*, have been well-studied and can be quite accurately predicted for any new alloy. One may conclude that for significant advances in the above respects, we should examine the prospects of alternative processes, for example, electron beam melting where differences in process chemistry and heat balance could be used to overcome the known limitations of VAR.

2.3. Electroslag Remelting

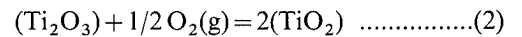
The electroslag remelting process, originally devised as a controlled solidification process for superalloys was expanded into general application as a de-sulphurizing technique for alloy steels. Superseded in that role by the more economic ladle-steelmaking methods, it is again being recognized that the process can be used with advantage in superalloys. The principal differences between ESR and VAR lie in the more complex ESR chemical system and in the slightly different thermal regimes.¹⁷⁾

The chemical regime differs from VAR in that the system can react with air, and that slag/metal reactions are also possible. The system is very far from simple thermochemical equilibrium. The mass flows are high and the system contains not only temperature gradient effects, but also significant electrochemical reactions. The reaction scheme has been considered in detail in very few systems and, unfortunately, none of them are superalloys.¹⁸⁾ However, we may draw some interesting conclusions from those studies.

It appears that the main reaction site in the system is the electrode/slag interface. The combination of residence time and surface/volume ratio renders this site more active than either the falling droplets or the ingot/slag interface. As a result, for the redox reactions of, for example,

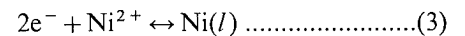


which control the Al/Ti balance in the superalloys are essentially complete when the metal leaves the electrode. Since this reaction site is very close to the slag/air surface, at which we have a second reaction:

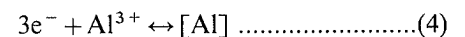


it is obviously important to maintain a stable electrode/slag immersion to obtain a satisfactory composition control. In this context, two additional items should be considered. Although we can eliminate reaction (2) by excluding air, for example, by argon, reaction (1) will still control the Al/Ti ratio in the ingot. Additionally, we should note that such slags contain both Ti^{3+} and Ti^{4+} . This system permits a substantial electronic conductivity in the slags and so lowers the resistivity to a point at which a satisfactory ESR heat balance cannot be achieved. Superalloys which contain a high Ti/Al ratio are consequently very difficult to process by ESR.

The role of electrochemistry¹⁹⁾ in the system appears to be minimal in the case of Ni- and Co-base alloys, in contrast to its important role in steels. The corrosion/oxidation reactions:



or



which accommodate the Faradaic transfers across the metal/slag interfaces are evidently more highly reversible in superalloys than are their equivalents in steels. Probably for this same reason installations have been operated in direct current (in contrast to the normal line frequency) melting superalloys for less-demanding applications. The combination of the above considerations has shown that the forging grades of superalloys, with a moderate level of γ^1 and δ -forming elements, can be melted through ESR slags in which the composition balance can be maintained whilst still retaining all the remaining physical requirements of the slag system. In a case where only one reactive element is involved, for example in an aluminide alloy, the slag/metal reactions are easily accommodated by appropriate oxide addition to the slag.

The de-sulphurizing role of ESR is important in superalloy processing. The alloy requirements for adequate intermediate-temperature ductility dictate a very low sulphur content. In the past, we have attempted to compensate for high sulphur contents (10–20 ppm) by additions of magnesium. Unfortunately, not only is such a technique difficult to control,²⁰⁾ due to the oxidation and volatilization of Mg during VIM and VAR, but also Mg volatilization has a critical influence on arc behaviour in VAR to the point at which the drip-short technique can be invalidated by the day-to-day variations in Mg content in VIM-cast electrodes. An excellent alternative technique is to use CaO desulphurization²¹⁾ in VIM, followed by ESR. The ESR then serves the dual function of assuring the removal of any slag trapped in the VIM electrode, and also of accomplishing further desulphurization through the ESR slag/metal reactions. By this technique we can reliably produce superalloys of < 1 ppm

sulphur content which can be reproducibly melted by the final VAR stage.

The behaviour of oxide and nitride inclusions in ESR superalloys is quite well understood.²²⁾ The oxide inclusions in the electrode dissolve as it melts, either in the electrode metal, or in the liquid slag. In consequence, the liquid metal in the ingot pool contains no oxide inclusions which are relics of the electrode content. As the ingot metal cools and freezes, it precipitates inclusions which are the result of slag/metal reactions on the electrode. The final inclusion population in the ingot is therefore a characteristic of the ESR system and is not related to the prior VIM process. This reaction sequence is part of the reason that ESR has acquired the reputation of being a more "forgiving" process in respect to electrode quality than is VAR. In tests of both a statistical and a quantitative nature, ESR superalloys have been found to have a lower content of large oxide inclusions than has VAR. This finding is in agreement with the above mechanism.²³⁾

Nitride inclusions (TiN) do not dissolve in ESR slags. The TiN solution equilibrium:



applies throughout the process, causing partial solution in the electrode film and partial precipitation in the ingot according to the nitrogen content relative to TiN saturation solubility in the given superalloy. At high nitrogen contents relative to saturation (*e.g.*, 100 ppm N in IN718, TiN saturation at ~ 40 ppm) some TiN inclusions may be moved by surface tension forces to the ingot rim and subsequently frozen in the ingot surface. In IN718, for example, nitrogen contents as high as 300 ppm have been noted in the outer 1 mm of the ingot surface. By this mechanism, an ingot content of 100 ppm might be reduced to 80 ppm. However, there exists a slight solubility of Ca_3N_2 in ESR slags, which will transfer some nitrogen from an air atmosphere into a superalloy. For IN718, for example, this reaction appears to limit the attainable ingot nitrogen content to approximately 60 ppm. Without the use of an argon atmosphere alloys with nitrogen contents lower than this value cannot be melted by ESR. It is noteworthy in this content that reaction (5) is sensitive to Ti content in the alloy, and that the nitrogen activity is also an inverse function of chromium content. We find, therefore, that alloys with high Ti content and low Cr content present much more of a nitrogen control problem in ESR than does IN718.

The heat balance in ESR has been exhaustively studied. In the specific case of superalloys, it appears that the limits of solidification control are approximately the same as for VAR, in spite of the very different ingot boundary conditions. Also, since a major component of the ingot enthalpy flow is the melting rate, we find that the melting rates required at these limiting conditions in ESR are similar to those found in VAR. It is extremely difficult to operate an ESR process at low heat input. The slag temperature distribution needed to retain both a stable melting condition and also an acceptable slag-skin formation on the crucible surface falls within a very

narrow process window. The combination of control conditions in respect to melt-rate and electrode positioning required to manufacture ingots reliably to rotating part standards has so far largely defeated industrial practice. At the present time very few engine rotating-part specifications permit the use of ESR alloys. It should be emphasized in this respect that the application problem is not one of acceptable properties—the best ESR superalloy has been shown to be better than VAR in regard to LCF life—the present difficulty is that of process reproducibility and quality assurance, rather than optimum properties. In the case of the triplemelt practice, high ESR melt-rates are permitted since structure control is not at issue and, consequently, the control problem becomes much simpler.

Generally, we may state that the ESR process development is quite similar to that of VAR. It is a process with well-defined advantages and known drawbacks. It has a clear application in making clean superalloys provided that for the large-section rotating-part applications we can use some other subsequent technique to control the alloy's microstructure. Since this latter drawback has risen from the fundamental nature of the ESR heat balance, it is unlikely that we will be able to make any significant advances in ESR application using the presently-known form of the process.

3. Present and Future Development

The case for a new process can most easily be made for titanium alloys, where the hard- α defect persists through triple VAR to the extent that it is the single most serious problem at present facing the aircraft engine material producer. Developments over the past seven years have confirmed that cold-hearth melting/ingot casting using either electron-beam power or plasma torches can solve this problem,¹⁶⁾ and both are now in use to prepare electrodes for a final VAR melt to control ingot structure.^{24,25)} In part spurred by these developments, EB and plasma have now both become accepted industrial techniques, with capital and operating costs not too dissimilar to VAR and ESR. It is germane to speculate, therefore, on the future potential of the processes for not only titanium but also superalloys.

Although both EB and plasma appear to be successful in providing the correct thermal regime for the solution of hard- α in titanium, the real need in new processing beyond this application is for a process of controlled solidification. In this respect, it would appear that EB has the greater potential since it is a much more precise heat-source than is plasma. Once having uncoupled the ingot heating source from the melting process, and also removed the heat-shielding effect of a VAR (or ESR) electrode tip, we find that the EB ingot thermal regime is essentially a balance between the condensing beam energy and prompt thermal radiation from the ingot free surface.²⁶⁾ The effect of this situation is render the system free to adopt virtually any system of temperature gradients during solidification. It has been shown that even under "normal" melting conditions, the EB ingot

pool in the melting of titanium alloys is much shallower than that in VAR, with consequent structure advantages.²⁶⁾ The same reasoning may be applied to superalloys, with the potential that EB may offer the long-sought goal of fine as-cast structure in a large-diameter superalloy ingot. The advantages of such a scheme have already been demonstrated in the VADER process,²⁷⁾ but as yet have not been industrialized by this technique due to difficulties in the process development. An attractive feature of an EB route for turbine materials would be the high degree to which the process can be put under computer control and assessment which is far beyond that possible in VAR or ESR. On-line continuous temperature and composition monitoring are already in operation for the EB process.

A second potential area of growth for EB is that of inclusion removal from superalloys. The controlled environment of the cold-hearth permits inclusion removal by both flotation and chemical decomposition. This technique is especially effective with the more stable inclusion types (HfO₂ and TiN) found in expensive airfoil alloys. The practice is now qualified for the recycling of casting scrap in these alloys and will certainly expand to other, similar applications, for example in the casting process itself. It has been shown also for example, that the method has the potential to remove all oxide inclusions from stainless steels; a concept which, if extended to superalloys, would revolutionize the way in which we design turbines by eliminating all statistical variation in properties and hence removing the need for conservative life prediction based on fracture mechanics prescriptions.

In considering the above areas for growth in the processes, it is to be noted that most turbine material applications involve a fundamental requirement of vacuum processing, whether for ingot or casting use. Whilst it is not impossible to consider the use of plasma as a heat source in vacuum, production experience of such furnaces has historically not been encouraging.

4. Conclusions

The near-term future developments in the melting and casting of superalloys and titanium alloys for turbine use will be concentrated on refining the process control and monitoring of VIM, VAR and ESR, particularly in extending the application of triple melting. The growth of electron beam technology into these areas has already started, in titanium alloy production and in superalloy recycling. This tendency will continue to grow as the next generation of alloys comes into use. Plasma melting has a limited field of application, but will continue to be an alternative to EB in primary titanium production.

The recent trend towards quality assurance by process control rather than by product inspection will continue. User specifications will increasingly concentrate on aspects of process monitoring. This scheme will need close cooperation between user and producer to arrive

at a satisfactory result. A similar close cooperation between the end-user and the research community will also be necessary in these developments to avoid wasting research effort in areas where there is no likelihood of industrial application because the proposed process change cannot be satisfactorily controlled.

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