THE EFFECT OF SILICON SILICONSILICON ADDITIONS ADDITIONSADDITIONS ON THE THERMAL THERMAL STABILITY AND MORPHOLOGY OF CARBIDES IN A Ni-Mo-Cr SUPERALLOY

Li Jiang, Zhoufeng Xu, Zhijun Li, Yanling Lu, Guanyuan Wu, Xingtai Zhou

Shanghai Institute of Applied Physics, Chinese Academy of Sciences; 2019 Jia Luo Road, Jiading District; Shanghai, 201800, China

Keywords: Ni-Cr-Mo, silicon, carbides, thermal stability.

Abstract

The effect of silicon additions on the thermal stability and morphology of carbides has been investigated in a Ni-Mo-Cr superalloy. It is found that granular M6C carbides form at the grain boundaries in the standard heat. The size and morphology of M6C carbides remain unchanged in the aging treatment at 700-800 ℃ up to 1000 h. While in the non-silicon heat, the intergranular carbides are examined as M2C type carbides by TEM at the initial stage of the aging treatment. With the aging time increasing, the fine granular M_2C carbides transform into coarse planar M_6C carbides. It can be concluded that silicon additions can lead to the more stable intergranular carbides in the Ni-Mo-Cr superalloy.

Introduction

Hastelloy N is a Ni-Mo-Cr superalloy developed in Oak Ridge National Laboratory for Molten-Salt Reactor Program in the 1970s. This alloy contains 16%-17% molybdenum for strengthening and 7% chromium sufficient to impart moderate oxidation resistance in air but not enough to lead to high corrosion rates in salt. There is additional 0.5%-1% silicon in the airmelted heats of this alloy for deoxidants as compared to the vacuum-melted heats.

It was found that silicon additions play a more important role in the structure and postirradiation properties of Hastelloy N. R. E. Gehlbach and H. E. McCoy [1] found that the microstructure of Hastelloy N is characterized by stringers of massive primary M6C carbides in matrix. The M6C carbides in air-melted heats are enriched in silicon and are not dissolved at high annealing temperatures but melt and transform to a noncarbide phase. In vacuum-melted heats with low silicon contents, primary M_6C carbides go into solid solution. Enough experiments [5-9] suggested that helium generation from the (n, α) reaction of boron at grain boundaries causes the irradiation brittleness in Hastelloy N. The massive primary M_6C carbides in air-melted heats can effectively fix boron atoms in the matrix and decrease the quantity of helium forming at grain boundaries to improve the irradiation brittleness.

On the other hand, H. E. McCoy and R. E. Gehlbach [10] found that the additions of silicon have obvious effects on the morphology of intergranular carbides in Hastelloy N. In the airmelted heats, the fine M_6C carbides precipitate at the grain boundaries and remain stable over an irradiation temperature range of 650-760 ℃. These fine precipitates may make it more difficult for cracks to propagate along the grain boundaries and increase the resistance to the intergranular fracture. The vacuum-melted heats form M2C carbides whose size and morphology depended

markedly upon the irradiation temperature. Irradiation at 760 ℃ results in coarser-dispersions of
the M₂C carbides and inferior postirradiation properties. So, it can be conclude that the carbides
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the Ms-C carbides and inferior postirradiation properties. So, it can be conclude that the carbides
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distribution (or the content of silicon) is of importance in two respects: (a) it will likely
determine he distribution distribution (or the content of silicon) is of importance in two respects: (a) it will likely
determine the distribution of helium, and (b) it will determine how easily cracks can propagate.
He potential effect of differe determine the distribution of helium, and (b) it will determine how easily cracks can propagate.

However, the content of silicon is not the only variable in these studies mentioned above.

The potential effect of differe However, the content of silicon is not the
The potential effect of different melting meth
neglected. In this paper, one standard vacuum-m
vacuum-melted heat are used to investigate the
morphology of carbides in Ni-Mo-Cr su **Experimental metallical constant in the space of the standard vacuum-melted heat will alta will alta are used to investigate the effect of simplodgy of carbides in Ni-Mo-Cr superalloy.

Experimental procedure**

As shown morphology of carbides in Ni-Mo-Cr superalloy.
 Experimental procedure

As shown in Table I, the Nominal chemical composition (wt. %) of the two heats is

identical to that of the standard Hastelloy N. The process for f **Experimental procedure**
 Experimental procedure

identical to that of the standard Hastelloy N. The process for fabrication of the wrought alloy is

as follows. The cast materials were propered by vacuum induction melt

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Alloy Heat		Mo		Fe	Mn		
Standard heat					U.)	0.5	0.05
non-silicon heat							

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anneal in heat treating furnace at 117 ingots were hot forged into 20mm-in-diameter bars. All samples were cut from
annealed in heat treating furnace at 1177 °C for 0.5h, followed by water
Subsequently, the samples were exposed at 700, 750 and 800 °C for 1-1000 annealed in heat treating furnace at 1177 °C for 0.5h, followed by water quenched

Subsequently, the samples were exposed at 700, 750 and 800 °C for 1-1000 h, respectively

Table 1: Nominal Chemical Composition of Ni-Mo-C Subsequently, the standard heat

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Table I: Nominal Chemical Composition of Ni-Mo-Cr Alloy.

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and polishing techniques and etched with the mixed solution (3g CuSO₄+10ml H₅SO₄+40ml

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and polishing techniques and etched with the mixed soluti **IDENTIFY IS THE UNITE THE METATIFY THE METATIFY ON THE MET AND THE MET AND THE MET AND HE MET AND HE MET AND HE MICH THOTS ON THE MICROSOPE (OM) and scanning electron microscope (SEM, LEO 1530VP) with energy dispersive s**

Metallographic samples were prepared using standard metallographic mechanical grinding
and polishing techniques and etched with the mixed solution (3g CuSO₄+10ml H_SO₄+40ml
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microscope (OM) and scanning electron micr and polishing techniques and etched with the mixed solution (3g CuSO₄+10ml H2₅SO₄+40ml
HCl⁺⁴50ml Water) to reveal the microstructure. The microstructures were examined by optical
microscope (OM) and scanning elect HCI+50ml Water) to reveal the microstructure. The microstructures were examined by optical polishing electron microscopy (SM) and scanning electrom microscopy (SEM, LEO 1530VP) with emais dispersive spectrometre (EDS) ana Interoscope (OM) and scaling electron interoscope (Adispersive spectrometre (EDS) analysis. Furthermore, transition tecnal G2 F20 S-TWIN) was used for phase identification electrochemical polishing at a solution of 5% perc Microstructure of Solution-Treated Ni-Mo-Cr Superalloy
 Results and discuss

Microstructure of Solution-Treated Ni-Mo-Cr Superalloy

The microstructure observations of Ni-Mo-Cr superalloy

The microstructure observation **Results and discussion**
Microstructure of Solution-Treated Ni-Mo-Cr Superalloy
The microstructure observations of Ni-Mo-Cr superalloy
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shown in Fig. Microstructure of Solution-Treated Ni-Mo-Cr Superalloy solution-treated at 1177 \degree for 0.5

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shown in Fig. 1. It can be seen The microstructure observations of Ni-Mo-Cr superalloy solution-treated at 1177 \degree for 0.5 h are shown in Fig. 1. It can be seen that the microstructure consists of equiaxed grains. As shown in Fig. 1a, there are many s The microstructure observations of Ni-Mo-Cr superalloy solution-treated at 1177 °C for 0.5
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shown in Fig. 1a, there are many stri h are shown in Fig. 1. It can be seen that the microstructure consists of equiaxed grains. As
shown in Fig. 1a, there are many stringers of primary M₆C carbides along the longitudinal
direction in the standard heat. Whil shown in Fig. 1a, there are many stringers of primary M_6C carbides along the longitudinal
direction in the standard heat. While the stringers in the non-silicon heat almost decompose into
the matrix and only a small qu direction in the standard heat.
the matrix and only a small grains or at the grain boundar
it can be inferred that there at
than that of the standard heat.

heat.

Figure 1. Microstructures of Ni-Mo-Cr superalloy in (a) the standard heat and (b) the non-silicon
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ging treatment and 750 CV Figure 1. Microstructures of Ni-Mo-Cr superalloy in (a) the standard Heats.
The Precipitation Behavior of M₆C Carbides in Standard Heats.
Fig. 2 shows the progressive morphological changes of integral and the pattern in Fig. 2 shows the processive morphological changes of intergranular precipitates under the aging treatment at 750 °C with increasing aging time. The selected area electron diffraction pattern in Fig. 3 indicates that the in The Precipitation Behavior of M_oC Carbides in Standard Heats

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aging treatment at 750 °C with increasing aging time. The select The Precipitation Behavior of M₆C Carbides in Standard Heats
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aging treatment at 750 °C with increasing aging time. The selected Fig. 2 shows the progressive morphological changes of intergranular precipitates under the aging treatment at 750 °C with increasing aging time. The selected area electron diffraction pattern in Fig. 3 indicates that the Fig. 2 shows the progressive morphological changes of intergranular precipitates under the aging tracturent at 730 C with increasing aging time. The selected area electron diffraction after any carbides and the EDS analys aging treatment at 750 °C with increasing aging time. The selected area electron diffraction
pattern in Fig. 3 indicates that the intergranular perceiptiates are M₆C carbides, and the BDS
analyses in Fig. 3(c) show that pattern in Fig. 3 indicates that the intergranular precipitates are M_6C carbides, and the EDS
analyses in Fig. 3(c) shown that the M_6C carbides contain Ni and Mo as the main metal atoms. As
shown in Fig. 2(a), the g

analyses in Fig. 3(c) show that the M₆C carbides contain Ni and Mo as the main metal atoms. As
shown in Fig. 3(c), the grain boundaries without any carbides are observed after the solution
treatment. And as aging time i shown in Fig. 2(a), the grain boundaries without any carbides are observed after the solution
treatment. And as aging time increases to 100 h, it is observed that the granular M₆C carbides remain unchanged during aging The tractment. And as aging time increases to 100 h, it is observed that the granular M₆C carbides
form at grain boundaries. The size of M₆C carbides is 200 nm. The morphology and size of the colution process
granular form at grain boundaries. The size of M₆C carbides is 200 nm. The morpholog
granular M₆C carbides remain unchanged during aging treatment up to 1000 h
 $2(c)$ and (d). Also, the density of granular M₆C carbides at 700 described at 750 °C.
The Precipitation and Decomposition Behavior of Carbides in Non-silicon Heats
The microstructure observations of the non-silicon heat exposed at 700-800 °C for 1-500 h
are shown in Fig. 4. It can be f The Precipitation and Decomposition Behavior of Carbides in Non-silicon Heats

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The microstructure observations of the non-silicon heat exposed at 700-800 °C for 1-500 h
are shown in Fig. 4. It can be found that granular pr The microstructure observations of the non-silicon heat exposed at 700-800 °C for 1-500 h
are shown in Fig. 4. It can be found that granular precipitates form densely at almost all grain
boundaries at the initial stage of The microstructure observations of the non-silicon heat exposed at 700-800 °C for 1-500 hare shown in Fig. 4. It can be found that granular precipitates form densely at almost all grain and specified as M₂C carbides and are shown in Fig. 4. It can be found that granular precipitates form densely at almost all grain
boundaries at the initial stage of aging treatment as shown in Fig. 4(a), (d) and (c). These fine
spranular precipitates are boundaries at the initial stage of aging treatment as shown in Fig. 4(a), (d) and (c). These fine
granular precipitates are identified as McC carbides and consist mostly of Mo, C and C atoms as
shown in Fig. 5. The size o granular precipitates are identified as M₂C carbides and consist mostly of M₀, Cr and C atoms as shown in Fig. 5. The size of granular M₂C carbides is 100 nm. When specimes are exposed for all onger time, the fine g graphin Fig. 5. The size of granular M₂C carbides is 100 nm. When specimens are exposed for
a longer time, the fine granular M₂C carbides dissolve from a portion of grain boundaries and
then the planar precipitates fo a longer time, the fine granular M_2C carbides dissolve from a portion of graitent the planar precipitates form as shown in Fig. (b), (e) and (h). The planar identified as MC carbides by the diffraction pattern in Fig. then the planar precipitates form as shown in Fig. (b), (e) and (h). The planar precipitates are identified as McC carbides by the diffraction pattern in Fig. 6. It can be observed that the fine granular McC carbides are

identified as M₆C carbides by the diffraction pattern in Fig. 6. It can be observed that the fine
granular M₂C carbides are replaced completely by coarse planer M₆C carbides at grain
boundaries at the final stage of granular M₂C carbides are replaced completely by coarse planar M₆C carbides at grain boundaries at the final stage of aging treatment as shown in Fig. 4(c), (f) and (i).

From Fig. 4 it can also be found that the sque boundaries at the final stage of aging treatment as shown in Fig. $4(c)$, (f) and (i).

From Fig. 4, it can also be found that the sequential evolution of the morphology of graboundaries with the aging time increasing. At

is related closely to the morphology of the intergranular carbides in other alloys $[11-12]$. The planar carbides.

Figure 6. TEM analysis of intergranular M₆C carbides in the non-silicon heat at 750°C for 100 h:
(a) microstructure observation and (b) corresponding diffraction pattern and (c) the EDS analyses
is related closely to th Figure 6. TEM analysis of intergranular M_oC carbides in the non-silicon heat at 750°C for 100 h:

(a) microstructure observation and (b) corresponding diffraction pattern and (c) the EDS analyses

is related closely to Figure 6. TEM analysis of intergranular M_oC carbides in the non-silicon heat at (a) microstructure observation and (b) corresponding diffraction pattern and (c) is related closely to the morphology of the intergranular The Example of the numerical state of the same of the same of the Schemetra and C interpretation of the EDS analyses
the intergranular carbides in other alloys $^{[11-12]}$. The planar
adjacent grain and push the local boun Figure 6. TEM analysis of intergranular M₆C carbides in the non-silicon heat at 750°C for 100 h:
(a) microstructure observation and (b) corresponding diffraction pattern and (c) the EDS analyses
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is related closely to the morphology of the intergranular carbides in other alloys $[11-12]$. The plana
carbides grow from is related closely to the morphology of the intergranular carbides in other alloys $^{[11-12]}$. The planar carbides grow from one grain into the adjacent grain and push the local boundary segment to cause serrations. The s carbides grow from one grain into the adjacent grain and push the local boundary segment to cause serrations. The same process occur in this alloy during the growth of intergranular M_sC carbides.
It can be found that th cause serrations. The same process occur in this alloy during the growth of intergranular M₅C carbides in the non-silicon heat at the initial stage of the aging treatment. This fact probably results from the enrichment It can be found that the precipitation of M_2C carbides only occur at the grain boundaries in the ano-silicon heat at the initial stage of the aging treatment. This fact probably results from the enrichment of carbon at It can be found that the precipitation of M₂C carbides only occur at the grain boundaries in
the non-silicon heat at the initial stage of the aging treatment. This fact probably results from the
forcincle matrices of ca the non-silicon heat at the initial stage of the aging treatment. This fact probably results from the enrichment of carbon atoms at the grain boundaries in the non-silicon heat. The massive primary MGC carbides are releas enrichment of carbon atoms at the grain boundaries M_6C carbides dissolve during the solution treatt carbides are released into the matrix and increase the matrix, especially at grain boundaries. Reseed islocations, sta dislocations, stacking faults and grain boundaries [13]. Therefore, the nucleation and subsequent ent of carbon atoms at the grain boundaries in the non-silicon heat. The massive primary
bides dissolve during the solution treatment. Subsequently, carbon atoms from M_6C
are released into the matrix and increase the s M₆C carbides dissolve during the solution treatment. Subsequently, carbon atoms from M₆C carbides are released into the matrix and increase the supersaturation degree of carbon atoms in the the matrix of segregation, carbides are released into the matrix and increase the supersaturation degree of carbon atoms in
the matrix, especially at grain boundaries. Research indicates that carbon atoms segregate to
dislocations, stacking faults

the matrix, especially at grain boundaries
dislocations, stacking faults and grain boun
growth of M₂C carbides are promoted by cat
the primary M₆C carbides in the standard
atoms for the precipitation of M₂C carbides dislocations, stacking faults and grain boundaries ^[13]. Therefore, the nucleation and subsequent
growth of M₂C carbides are promoted by carbon segregation at the grain boundaries. In contrast,
the primary M₆C carbi growth of M₂C carbides are promoted by carbon segregation at the grain boundaries. In contrast, the primary M₆C carbides in the standard heat remain stable, and there are not enough carbon atoms for the prequitation o the primary M₆C carbides in the standard heat remain stable, and there are not enough carbon stor the precipitation of M₂C carbides.
While the M₂C carbides are relatable and decomposes with the aging time increasing atoms for the precipitation of M₂C carbides.

While the M₃C carbides are metastable and decomposes with the aging time increasing at

700-800 °C, and then the planar M₆C carbides form at grain boundaries. It never b While the M₂C carbides are metastable and decomposes with the aging time increasing at 700-800 °C, and then planar M₆C carbides form at grain boundaries. It never be found that the escond M₆C carbides are close to M 700- 800 °C, and then the planar M_6C carbides form at grain boundaries
the second M_6C carbides are close to M_2C carbides at the same grain b
fact indicates that the M_2C carbides and the precipitation of M_6C

$$
(Mo, Cr)_2C \rightarrow 2Me(Mo, Cr) + C \tag{1}
$$

$$
6\text{Me(Ni, Mo)} + C \rightarrow (\text{Ni, Mo})_6C \tag{2}
$$

(Mo, Cr)₂C → 2Me(Mo, Cr) + C (1)
 $6Me(Ni, Mo) + C \rightarrow (Ni, Mo) \& C$ (2)

The instability of M₂C carbide probably results from the high content of Cr as shown in Fig

5(c). A first-principles study ¹¹⁴ on the structural of Mo₂ (Mo, Cr)₂C → 2Me(Mo, Cr) + C

6Me(Ni, Mo) + C → (Ni, Mo)₆C

(2)

The instability of M₂C carbide probably results from the high content of Cr as shown in Fig

5(c). A first-principles stdy ¹⁴⁴¹ on the structural of (Mo, Cr): $C \rightarrow 2Me(Mo, Cr)+C$ (1)

6Me(Ni, Mo) + C \rightarrow (Ni, Mo)₆C (2)

The instability of M₂C carbide probably results from the high content of Cr as shown in Fig.

5(c). A first-principles study ^[14] on the structural $6\text{Me(Ni, Mo)} + C \rightarrow (Ni, Mo) \cdot C$ (2)

The instability of M_2C carbide probably results from the high content of Cr as shown in Fig. 5(c). A first-principles study ^[14] on the structural of Mo₂C has proved that the formation 6Me(Ni, Mo) + C \rightarrow (Ni, Mo)₈C (2)

The instability of M₂C carbide probably results from the high content of Cr as shown in Fig.

5(c). A first-principles study ^[14] on the structural of Mo₂C bas proved that the The instability of M₂C carbide probably results from the high content of Cr as shown in Fig.
5(c). A first-principles study ¹¹⁴¹ on the structural of Mo₂C has proved that the formation
enthalpies of Hexa-Mo_CC and The instability of M₂C carbide probably results from the high content of Cr as shown in Fig. 5(c). A first-principles study ¹¹⁴¹ on the structural of Mo₂C has proved that the formation of granular M₂C carbides at 5(c). A first-principles study ^[14] on the structural of Mo₂C has proved that the formation enthalpies of Hexa-MoCrC gradter and stable carbidele, as shown in Fig. 4, the critical time for the decomposition of granula enthalpies of Hexa-MocC and Hexa-MoCrC are -0.34 and -0.25 eV per unit cell, which indicates
that the Hexa-MoCC sytem is relatively instable. As shown in Fig. 4, the critical time for the
decomposition of granular M_2C that the Hexa-MoCrC system is relatively instable. As shown in Fig. 4, the critical time for the decomposition of granular M₂C carbides at 700, 750 and 800°C are 500, 200 and 100 h, decomposition process is diffusion co

Conclusions Conclusions Conclusions

decomposition of granular M₂C carbides at 700, 7:
respectively. The M₂C carbides are more instable at a
decomposition process is diffusion controlled. The
diffuse more rapidly at grain boundaries at the h
decompositio respectively. The \overline{M}_2 C carbides are more instable at a higher aging temperature. This implies the decomposition process is diffusion controlled. The atoms dissolved from the M2C carbides diffuse more rapidly at grai decomposition process is diffusion controlled. The atoms dissolved from the M₂C carbides
diffuse more rapidly at grain boundaries at the higher aging temperature. And then the
decomposition process can finish within a s diffuse more rapidly at grain boundaries at the higher aging temperature. And then the decomposition process can finish within a shorter time.

During the decomposition process, the atoms such as Mo and C are released fro decomposition process can finish within a shorter time.

During the decomposition process, the atoms such as Mo and C are released from M₂C

carbides. These atoms contribute to the nucleation, growth and coarsening of s During the decomposition process, the atoms such as Mo and C are released from M₂C
carbides. These atoms contribute to the nucleation, growth and coarsening of second M_oC
grain boundaries almost remain the same with t carbide. The
carbides at grain boundar
such stable i
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Stable inter **Conclusions**

In the standard heat, the size and morphology of intergranular granular M₆C carbides

remain unchanged in the aging treatment at 700-800°C up to 1000 hours. In the non-silicon heat,

the fine intergranula **Conclusions**
 Conclusions

remain unchanged in the aging treatment at 700-800°C up to 1000 hours. In the non-silicon heat

the fine intergranular granular M2C carbides formed at the initial stage of the aging treatment **Conclusions**

In the standard heat, the size and morphology of intergranular granular M₆C carbides

term intergranular manuar M₂C carbides formed at the initial stage of the aging treatment at

700-800 °C. With the ag In the standard heat, the size and morphology of intergranular granular M₂C carbides
remain unchanged in the aging treatment at 700-800 °C up to 1000 hours. In the non-silicon heat,
the fine intergranular granular M₂C Inthe standard heat, the size and morphology of intergranular granular McC carbides

remain unchanged in the aging treatment at 700-800[°]C. With the aging treatment at 700-800[°]C. With the aging time increasing, the gr

Acknowledgments Acknowledgments

remain unchanged in the aging treatment at 700-800°C up
the fine intergranular granular M₂C carbides formed at the
700-800°C . With the aging time increasing, the granula
coarse planar M_oC carbides at grain boundaries. tostable intergranular carbides in the Ni-Mo-Cr superalloy.
 Acknowledgments

This work was supported by Science and Technology Commission of Shanghai Municipality

(Grant No. 11JC1414900), Project supported by the Nati **Acknowledgments**

(Grant No. 11JC1414900), Project supported by Thorium Molten Salts Reactor Fund (Grant No. 201000000), Project supported by the National Natural Science Foundation of China (Grant No. 11005148), Project **Acknowledgments**
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(Grant No. 111C1414900), Project supported by Thorium Molten Salts Reactor Fund (Grant No.

XDA020000000), Project supported by the National Natural Science, Poundation of China (Grant This work was supported by Science and Technology Commission of Shanghai Municipality (Grant No. 11JC1414900), Project supported by the National Natural Science Foundation of China (Grant No. 2005148), Project supported by This work was supported by Science and Technology Commission of Shanghai Municipality

(Grant No. 11/01-1414900), Project supported by the National Natural Science Foundation of China (Grant No. 11005148), Project supporte (Grant No. 11JC1414900), Project supported by Thorium

XDA02000000, Project supported by the National Nat

No. 11005148), Project supported by the Special P

Academy of Science, China (Grant No. 2010CB934501).

Research Pr

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